Colegio de Postgraduados

Citrus waste hydrochar performance as soil amendment in constructed wetlands for the treatment of wastewater from the sugarcane industry

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ABSTRACT

Objective: This study evaluated the performance of citrus waste hydrochar as a soil amendment in constructed wetlands for treating sugarcane industry wastewater, assessing its impact on organic matter removal and plant development.

Design/Methodology/Approach: Two horizontal subsurface flow constructed wetlands were compared: C1 (amended with orange waste hydrochar) and C2 (control). The systems treated diluted sugarcane wastewater (1 g COD/L) with a 72-hour hydraulic retention time over 150 days. Three plant species (*Canna indica*, *Spathiphyllum wallisii*, *Typha latifolia*) were monitored in triplicate for stem growth, leaf count, leaf length, and leaf width. Statistical analysis included Shapiro-Wilk normality tests and Mann-Whitney U comparisons.

Results: The hydrochar system showed initial negative removal efficiencies (−31.5% COD, −39.4% soluble COD) due to leaching phenomena, but outperformed the control during maturation (69.3% *vs.* 65.5% COD removal). Strong linear correlations between total and soluble COD were observed ($R^2=0.997$ C1, $R^2=0.983$ C2). Plant responses were species-specific: *Typha latifolia* showed 142.0% greater leaf length ($p=0.0124$), *Spathiphyllum wallisii* increased stem length by 31.9% ($p=0.0088$), while *Canna indica* exhibited trade-offs between stem growth and leaf production.

Limitations/Implications: Single-replicate wetland design limits statistical power for system-level comparisons. Initial hydrochar leaching requires pre-treatment strategies. Findings support circular-economy approaches through the valorization of agricultural waste.

Conclusion: Citrus waste hydrochar enhances constructed wetland performance after stabilization, particularly improving soluble COD removal and promoting selective plant growth, making it a promising amendment for wastewater treatment applications.

Keywords: Hydrothermal carbonization, constructed wetlands, agro-industrial wastewater, phytoremediation, circular economy, organic matter removal.

Citation: Landeta-Escamilla, O., Sandoval-Gonzalez, O. O., Alvarado-Lassman, A., Vallejo-Cantu, N. A., & Gonzalez-Serrano, A. (2025). Citrus waste hydrochar performance as soil amendment in constructed wetlands for the treatment of wastewater from the sugarcane industry. *Agro Productividad*. https://doi.org/10.32854/t440cz26

Academic Editor: Jorge Cadena Iñiguez
Associate Editor: Dra. Lucero del Mar Ruiz Posadas
Guest Editor: Daniel Alejandro Cadena Zamudio

Received: July 9, 2025.
Accepted: November 22, 2025.
Published on-line: December 11, 2025.

Agro Productividad, 18(11). November. 2025. pp: 219-239.

INTRODUCTION

Biomass conversion is crucial for waste recovery, as it transforms waste into valuable materials that support the circular economy. Biomass is typically categorized by its source: agricultural, forestry, municipal, or industrial organic waste (Konstantinavicienė &

Vitunskiene, 2023). In Mexico, citrus fruits are among the most widely produced crops, totaling 7.6 million tons (SIAP, 2019), with 51% of the oranges grown in Veracruz (USDA, 2023). One approach to biomass valorization is thermochemical processing. Hydrothermal carbonization (HTC) is a thermochemical method where biomass with high moisture content is subjected to low temperatures (180 °C-250 °C) and hot, pressurized water, resulting in a carbonaceous material called "hydrochar" (Islam, Limon, Romic, & Isam, 2021). Orange biomass is composed of 22% cellulose and 11% hemicellulose, among other components (Ayala *et al.*, 2021), which convert during HTC into hydrochar (solid fraction), lignite-like products, gaseous compounds, and liquid residues. LewandHydrochar, a carbon-rich product, has numerous applications. One notable use is as a soil enhancer, due to its cation exchange capacity and hydrophilic properties, which promote nutrient exchange between the substrate and plants (Wang *et al.*, 2025) and help maintain, or even increase, soil organic carbon (SOC) stocks (Kambo & Dutta, 2015). However, the amount of hydrochar applied must be carefully planned due to its phytotoxic effects (Celletti *et al.*, 2021); therefore, it should be mixed with other substrates to mitigate these effects.

Constructed wetlands (CWs) are eco-technologies designed to treat wastewater by emulating key features of natural wetlands (NWs), including phytoremediation and the use of substrates that mimic soil, plants, water, and bacterial communities. These elements work together to perform processes such as filtration, sedimentation, phytoaccumulation, and bacterial growth. To improve the effectiveness of CWs, various plant species are being tested for their ability to remove pollutants (Vymazal, J., 2022). Adding hydrochar to these systems is expected to enhance retention of suspended solids and promote biofilm formation by microorganisms that degrade organic matter.

Sustainable management of industrial wastewater is one of the most significant environmental challenges of the 21st century, particularly in regions with significant agricultural and industrial activity. The sugarcane industry, a key economic pillar in many tropical and subtropical regions, generates substantial volumes of effluents with high organic loads, nutrients, and suspended solids. Without proper treatment, these effluents can cause eutrophication, decreased dissolved oxygen levels, and alterations to the receiving aquatic ecosystems. In response to this problem, constructed wetlands (CW) have emerged as a passive treatment technology that combines purification efficiency with environmental and economic sustainability.

Constructed wetland systems mimic the natural purification processes of wetlands, using complex interactions between substrates, macrophytes, and microbial communities to remove contaminants through physical, chemical, and biological mechanisms. Sundaravadivel and Vigneswaran (2001) were among the first to highlight the potential of this technology as an alternative to conventional, energy-intensive systems, particularly in developing countries. More recent studies have confirmed that CW can achieve removal efficiencies of 70-98% for chemical oxygen demand (COD) and 80-99% for biochemical oxygen demand (BOD5) in horizontal subsurface flow configurations (Waly *et al.*, 2022; Nurmahomed *et al.*, 2022).

The optimization of these systems has led to the exploration of innovative materials to improve the filter medium. In this context, carbonaceous materials derived from agricultural waste have gained notable prominence. Biochar, produced by pyrolysis, and hydrochar, obtained by hydrothermal carbonization (HTC), represent biomass recovery strategies that align wastewater treatment with the principles of the circular economy (Escudero-Curiel *et al.*, 2023). Zhang and Tao (2022) comprehensively documented the unique properties of hydrochar for the adsorption of heavy metals such as Cr(VI) and Cd(II), attributing them to its developed porous structure and abundant oxygenated functional groups. Simultaneously, Marx and van der Merwe (2021) demonstrated that hydrochars derived from paper sludge can achieve phenol removal efficiencies comparable to those of activated carbon, with the added value of waste recovery.

The incorporation of these carbonaceous materials into constructed wetlands has revealed multifaceted benefits. Jagaba *et al.* (2023) reported that activated oil palm hydrochar in activated sludge reactors achieved removals of 84.66% for BOD5 and 72.07% for COD, while Aylan *et al.* (2023) observed significant improvements in BOD5 and ammonium removal in horizontal wetlands using palm biochar as a filter medium. Beyond improving purification, these materials serve as soil amendments that enhance the substrate's physicochemical properties. Lavagi *et al.* (2024) quantified the benefits of adding biochar to citrus nurseries, including improved nutrient availability, enhanced water retention, and increased plant growth, resulting in substantial economic benefits. Yu *et al.* (2020) confirmed that hardwood and bagasse biochars can replace up to 70% of conventional substrates without negatively affecting the growth of tomatoes and basil.

The selection of plant species is another crucial factor in determining CW performance. Licata *et al.* (2019) demonstrated that *Arundo donax* and *Typha latifolia* significantly outperform *Cyperus alternifolius* in contaminant removal, while polyculture systems were more efficient than monocultures for dissolved organic compounds. Zamora *et al.* (2019) evaluated ornamental species, including *Canna indica*, *Cyperus papyrus*, and *Hedychium coronarium*, and found 20-60% higher removals in planted microcosms compared to unplanted ones, in addition to identifying the commercial potential of these species.

Despite these advances, significant knowledge gaps remain. There is limited research on the specific use of hydrochar derived from citrus waste in constructed wetlands, particularly for treating effluents from the sugarcane industry. Citrus waste, generated in large volumes by the juice industry, represents an underutilized biomass source with the potential to produce high-quality hydrochar. Studies such as those by Palapa *et al.* (2024) have characterized hydrochars from fruit peels, revealing uniform and aggregated morphologies, although with smaller surface areas than materials derived from rice husks. Thawornchaisit *et al.* (2021) demonstrated that cation modifications, especially Fe^{3+}, can significantly enhance bagasse hydrochar's phosphorus removal capacity, suggesting ways to optimize the performance of materials derived from agro-industrial waste.

The integration of these lines of research, CW optimization, waste valorization through HTC, and improvement of the filter medium with carbonaceous material converges in the opportunity to develop more efficient and circular treatment systems. This study aims to contribute to the emerging field by evaluating the performance of hydrochar derived from orange solid waste (OSW) as a soil amendment in constructed wetlands with horizontal subsurface flow for wastewater treatment from the piloncillo industry. Specifically, the following were investigated: (1) the efficiency in removing organic matter (COD, BOD5) and total solids; (2) the effect of hydrochar on the growth of *Typha latifolia* and *Canna indica*; and (3) the significant differences in performance between systems with and without the addition of hydrochar. The results provide experimental evidence for the valorization of citrus waste and the development of more sustainable water treatment systems for the sugarcane industry.

Given the aforementioned, the present work evaluated the presence of citrus waste hydrochar in CW and its effect on plant growth to determine whether it improves CW behavior.

MATERIALS AND METHODS

This research employed a sequential and systematic methodology, beginning with the acquisition and preparation of solid orange waste (SOW) from the local market in Orizaba, Veracruz. The biomass, characterized by its physicochemical properties (total solids, volatile solids, moisture, COD, and pH), was converted to hydrochar via hydrothermal carbonization (HTC) at 180 °C, using pre-optimized parameters. The core of the study lay in the design, construction, and monitoring of two horizontal subsurface flow constructed wetlands, identical in dimensions and substrate stratification (tezontle and limestone layers), but with the experimental cell (C1) differentiated by the strategic inclusion of an SOW hydrochar layer. The system's performance in wastewater treatment and macrophyte development was evaluated over four months, operating with a 72-hour hydraulic retention time. The COD removal efficiency was statistically analyzed to determine the impact of hydrochar as a filtering medium. Figure 1 illustrates the development of the implemented methodology.

Obtaining and Preparing the Raw Material

Solid orange waste (SOW) was systematically collected from commercial establishments dedicated to citrus juice extraction, located within the Emiliano Zapata market in the

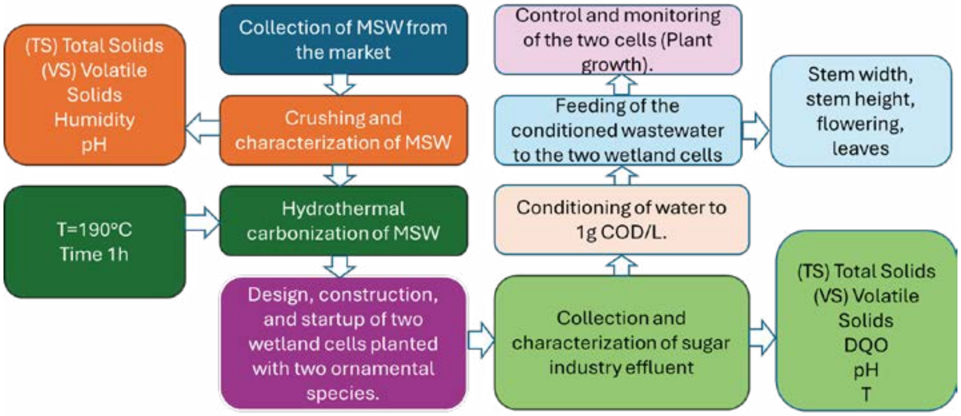


Figure 1. Methodology implemented.

city of Orizaba, Veracruz. Sampling was conducted in three independent batches during the fall season, thereby ensuring the temporal representativeness of the biomass. After collection, each batch was transported under controlled conditions to Environmental Engineering Laboratory I at the Technological Institute of Orizaba.

In the laboratory, a meticulous manual separation process was performed to remove inorganic materials and physical contaminants from the biomass. Once the OSWs were purified, dimensional reduction was performed by manual cutting, yielding homogeneous fragments of approximately 2 cm^2. Finally, the processed material was stored in sterile glass containers and kept at 4 °C to preserve its physicochemical properties until further processing. Figures 2 and 3 illustrates the process of collection and initial conditioning of the OSWs.

Figure 2. Process for collecting and conditioning orange solid waste (OSW).

Figure 3. Model CF1 Hydrothermal Carbonization Reactor.

Physicochemical Characterization of OSW

The physicochemical characterization of the three batches of OSW revealed parameters consistent with the typical composition of citrus waste (Table 1). Total solids (%TS) ranged from 19.86% to 21.20%, while volatile solids (%VS) ranged from 93.79% to 94.57%, indicating a high content of biodegradable organic matter. The moisture content exceeded 78%, confirming that OSW is a biomass with a high inherent moisture content, a favorable characteristic for hydrothermal carbonization processes.

Comparative analysis with previous research demonstrates the consistency of the parameters obtained. The pH values (3.89-4.38) are consistent with the characteristic acidity of citrus waste, as reported by Nava-Pacheco (2021) and Galván-Hernández (2024). The slight variation in %ST compared to the study by Muñoz-Valeriano *et al.* (2020) can be attributed to differences in the varietal composition and processing conditions of the biomass.

Determination of total solids (TS) and volatile solids (VS)

TS and VS were determined using the 2540 G Standard Methods gravimetric method. Each batch of RC collected was tested in triplicate. First, three crucibles were weighed to a constant weight. Then, 3 g of RC was added and placed in the drying oven for 24 hours at 105 °C. After 24 hours, they were removed from the oven and weighed. Finally, the crucibles were placed in a muffle furnace at 550 °C for 2 hours to calcine the sample, and the percentages of ST and SV were calculated.

Moisture determination

To determine the moisture content, the NMX-AA-16-1984 gravimetric method was employed, which involved weighing three crucibles to a constant weight and then adding 3 g of RC sample. The crucibles were placed in the drying oven for 24 hours at 105 °C.

Determination of Chemical Oxygen Demand (COD)

The COD parameter was determined using the 5220 D Standard Methods colorimetric method, which measures the amount of oxygen required to oxidize the organic matter in the effluent. The determination was carried out in a total (TCOD) and soluble (SCOD) manner. In the first step, the sample is stirred, and the analysis is carried out. In the second step, the sample is centrifuged to sediment the solids, and then the analysis is continued.

pH determination

The pH was determined using the NMX-AA-013-SCFI-2006 method, which involved weighing 10 g of RC in a beaker and adding 20 mL of distilled water, followed by stirring

Table 1. Parameters of the typical composition of citrus waste.

Lot	TS (%)	VS (%)	Humidity (%)	pH
1	20.32	94.49	79.67	3.89
2	19.86	93.79	80.13	4.38
3	21.20	94.57	78.79	4.13

for 30 minutes. Once stirring was complete, the mixture was left to stand for 15 min, and the pH was measured using an OAKTON potentiometer. The electrode was inserted into the sample, and the equipment was allowed to stabilize before the reading was taken.

Water conditioning

Water conditioning was carried out at an applied volumetric load (Cva) of 1 g COD/L to prevent ornamental plants from experiencing stress due to the high organic matter content in the effluent. The AVL was calculated using equation 4, which accounts for the sound volume of the cells, the feeding volume, and the concentration of organic matter determined by COD.

Wastewater feed

Once the water had been conditioned, it was fed into the two CW cells at a volume of 30 liters, with vertical flow to promote contact between the effluent and the substrates.

Hydraulic residence time

The hydraulic residence time is used to calculate the time that wastewater remains in contact with the substrates and plants. This parameter is important because contaminants are absorbed during the time that the water remains in the CW system.

pH determination

The pH was determined using the NMX-AA-013-SCFI-2006 method, which involved weighing 10 g of RC into a beaker, adding 20 mL of distilled water, and stirring for 30 minutes. Once stirring was complete, the mixture was left to stand for 15 min, and the pH was measured using an OAKTON potentiometer. The electrode was inserted into the sample, and the equipment was allowed to stabilize before the reading was taken.

Hydrothermal carbonization process

The transformation of OSW into hydrochar was carried out by hydrothermal carbonization (HTC) using a CF1 high-pressure reactor with a capacity of 1 L (Figure 4). The operating conditions were set at 180 °C with a residence time of 3 hours, parameters optimized from previous studies that demonstrate their effectiveness in maximizing yield and improving the physicochemical properties of hydrochar.

Operation and Conditioning

The system operated with a hydraulic retention time (HRT) of 72 hours, including a conditioning period of 14 days. Subsequently, it was fed with hydrolyzed piloncillo wastewater (ARP) at a concentration of 1 g COD/L, maintaining the established HRT.

Characterization and Conditioning of Wastewater

The mill effluent was collected in 20 L drums. The physicochemical characterization (Table 2) revealed a significant organic load (TOD: 301.2 g/L, TSS: 268.5 g/L), consistent

Table 2. Physicochemical characterization of sugar mill water.

Parameter	TS (%)	VS (%)	pH	COD (mg/L)	SCOD (mg/L)	Carbohydrates (g/L)
ARP	15.52	87.27	4.35	301.2	268.5	270

with that reported by Orduña-Gaytán (2023), although with variations in total and volatile solids attributable to seasonal and processing differences.

The concentration was adjusted to 1 g COD/L by calculated dilution using 0.833 L of ARP and 49.167 L of fresh water per feed cycle, optimizing the conditions for the development of the treatment system. The results obtained in this study on the use of citrus waste hydrochar as a soil amendment in constructed wetlands for the treatment of wastewater from the sugar cane industry reveal complex patterns in system performance.

Design and Construction of Constructed Wetlands

Two constructed wetland cells with horizontal subsurface flow (SSFH) were built with identical dimensions: 80 cm long × 30 cm wide × 30 cm high. The substrates were designed as follows:

Experimental Cell (C1): A vertically stratified configuration was implemented:

a) Layer 1 (inlet): Limestone, located at the beginning of the flow path to promote initial pH adjustment.
b) Layer 2 (middle): OSW hydrochar, contained in a mesh bag to prevent it from being carried away and clogging the system.
c) Layer 3 (top): Red tezontle, which served as the primary substrate for the anchoring and development of the plants' root system.
d) Layer 4 (outlet): Limestone, to ensure final stabilization of the effluent.

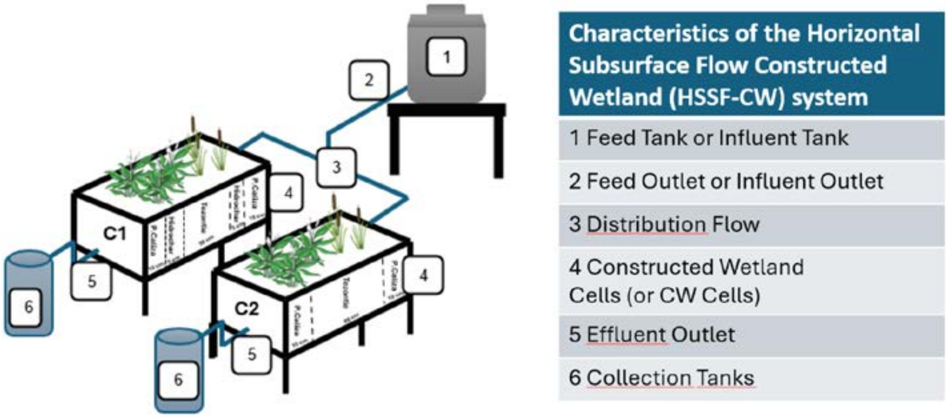
Control cell (C2): The same stratigraphic and material configuration as in C1 was replicated, except for the absence of hydrochar in the middle layer.

Figure 4 displays the individual components that constitute the HSSF-CW.

System Establishment and Operation

Each cell was planted in polyculture with two species of emergent macrophytes widely documented for CW: *Typha latifolia*, *Spathiphyllum wallisii*, and *Canna indica*. The plants were monitored for 4 months, with periodic evaluations of growth metrics, including stem height, leaf length, and shoot number.

The system was operated continuously with a hydraulic retention time (HRT) of 72 hours. Wastewater from the piloncillo production process was used as a synthetic influent. At the end of each HRT cycle, effluent samples were collected from both cells for analysis.



Characteristics of the Horizontal Subsurface Flow Constructed Wetland (HSSF-CW) system
1 Feed Tank or Influent Tank
2 Feed Outlet or Influent Outlet
3 Distribution Flow
4 Constructed Wetland Cells (or CW Cells)
5 Effluent Outlet
6 Collection Tanks

Figure 4. Design of the horizontal subsurface Flow Constructed Wetland system.

Figure 5. Wetland cells constructed in a polyculture configuration.

Control and monitoring of the two cells

To evaluate the vegetative development of ornamental plants, growth was recorded over four months, assessing the growth of leaves (number, width, and length), shoots (number), and stems (number, length, and width).

Statistical Analysis

The statistical analysis was designed to quantitatively evaluate the effect of citrus waste hydrochar as a soil amendment on both plant development and treatment efficiency within the constructed wetlands. The experimental unit for all analyses was the individual wetland cell (n=1 per treatment for system-level parameters, with plant metrics collected from multiple individuals within each cell).

Data normality and homoscedasticity were verified using the Shapiro-Wilk test and Levene's test, respectively. Given the sample size and experimental design, both parametric and non-parametric approaches were employed to ensure robustness.

The core of the analysis relied on Comparative Hypothesis Testing to determine the statistical significance of observed differences between the experimental cell (C1, with hydrochar) and the control cell (C2, without hydrochar). For plant growth parameters (Stem Length, Leaf Count, Leaf Length, Leaf Width) across the three species (*Canna indica*, *Spathiphyllum wallisii*, and *Typha latifolia*), unpaired two-sample t-tests were used for data meeting parametric assumptions. The results were considered statistically significant at a p-value <0.05.

The magnitude of the hydrochars effect was quantified by calculating the percentage difference for each metric. This allowed for a direct comparison of the relative improvement or worsening of each parameter.

To assess the efficacy of hydrochar, an Integrated Efficiency Index was calculated. This index assigned scientifically justified weights to the different plant metrics based on their importance in constructed wetland function: Leaf Length (0.4), Stem Length (0.3), Leaf Width (0.2), and Leaf Count (0.1). These weights reflect the greater relevance of larger leaf area and structural support for processes like contaminant adsorption, gas exchange, and system stability.

Finally, a robustness analysis was conducted. This involved evaluating not just the magnitude of improvement in individual metrics, but also the consistency of the positive effect across multiple metrics. A treatment was considered robust if it improved a majority of the measured variables, indicating a widespread physiological benefit rather than an isolated change.

All statistical calculations and analyses were performed using Python with the SciPy and Pandas libraries. The results are presented as mean values ± standard deviation where applicable, and significance is reported at the 95% confidence level.

RESULTS AND DISCUSSION

Treatment Efficiency and Temporal Behavior

The descriptive analysis showed average COD values of 1.315 mg/L for C1 (with hydrochar) and 0.596 mg/L for C2 (control), while soluble COD values were 1.339 mg/L and 0.60 mg/L, respectively. However, the removal efficiency values show particularly interesting behavior. System C1 showed negative average efficiencies for both total COD (−31.5%) and soluble COD (−39.4%), whereas the control system C2 showed positive efficiencies of 40.4% and 29.2%, respectively.

This apparent contradiction is explained by analyzing the temporal evolution of the systems. During the initial period (0-30 days), both systems showed negative efficiencies, with C1 (−212.0%) more pronounced than C2 (−20.7%). This phenomenon suggests an acclimatization period and possible leaching of hydrochar components during the initial stages of operation. The literature reports that carbonaceous materials, such as hydrochar, can release organic compounds during the early stages of implementation, which would explain the negative removal values observed. The temporal evolution of the organic matter concentration and its removal is shown in Figure 6. The figure comprises four panels monitoring the system performance over time: total and soluble

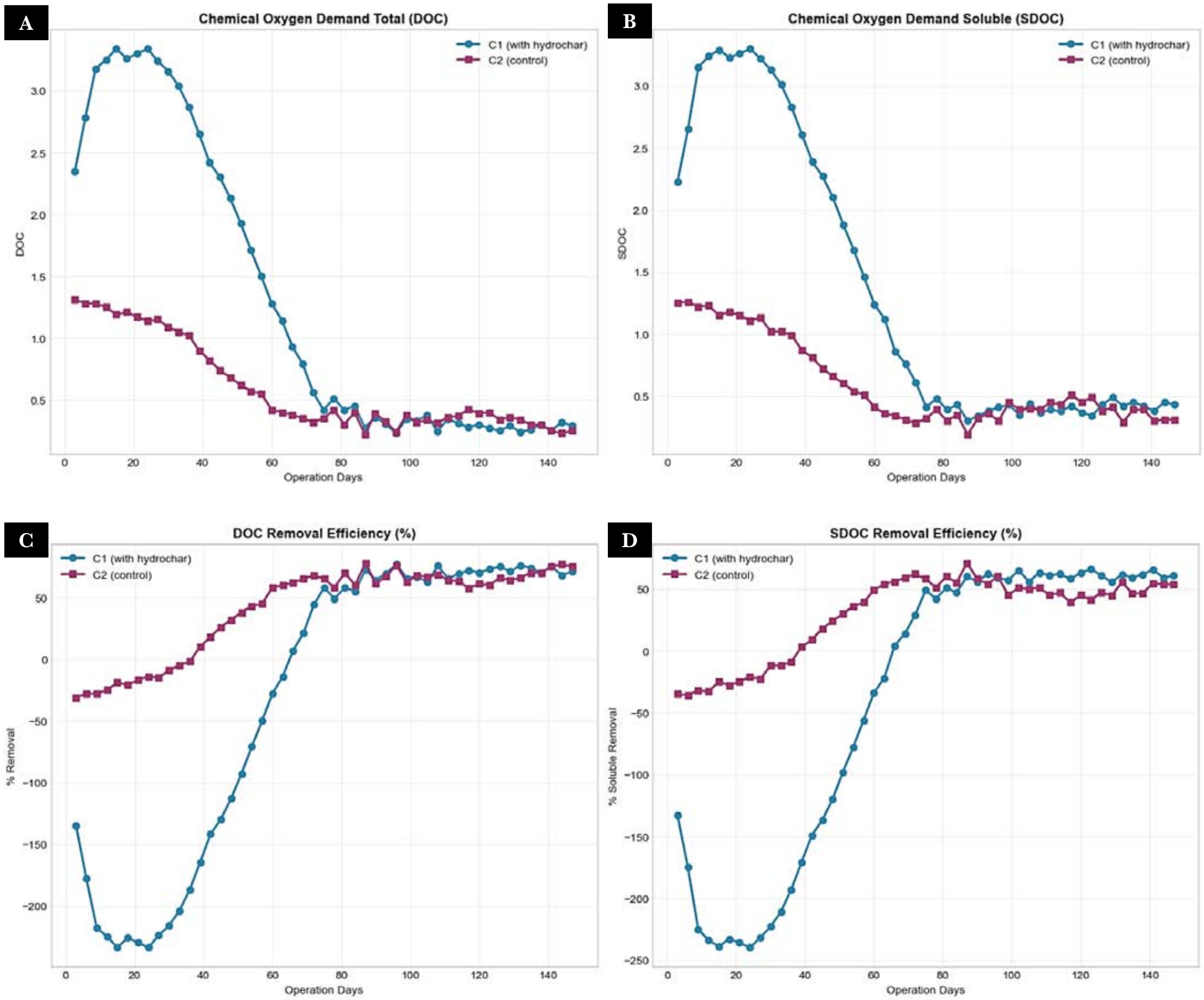


Figure 6. Temporal Analysis. A) Chemical Oxygen Demand Total (DOC), B) Chemical Oxygen Demand Soluble (SDOC), C) DOC Removal Efficiency, D) SDOC Removal Efficiency.

fractions of Chemical Oxygen Demand (COD and SCOD, panels A and B, respectively), along with their corresponding removal efficiencies (panels C and D).

System Stabilization and Maturation

The analysis by time period reveals a clear trend of improvement in the performance of both systems. While in the operation period (61-90 days), C1 reached an efficiency of 41.4% and C2 64.7%, during the maturation phase (91-120 days), C1 surpassed C2 with 69.3% versus 65.5%. This reversal in relative performance suggests that hydrochar requires a more extended stabilization period to realize its potential benefits.

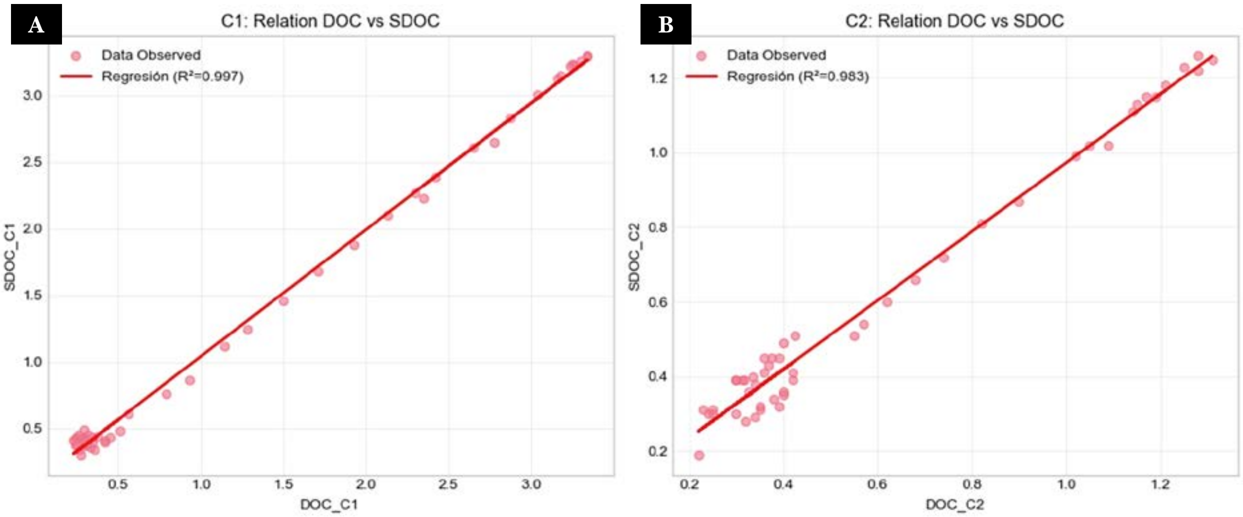


Figure 7. Linear Regression, A) Relation DOC *vs*. SDOC in C1 and B) Relation DOC *vs*. SDOC in C2.

The stable period (121-150 days) confirms this trend, with C1 showing slightly higher efficiency (72.6%) than C2 (69.2%). This pattern is consistent with previous studies indicating that carbonaceous materials require time to develop adapted microbial communities and to stabilize their surfaces before reaching their maximum treatment potential.

Linear regression analysis was performed to examine the relationship between total and soluble chemical oxygen demand in both systems. The results, presented below, are essential for understanding the proportional distribution of organic fractions and validating the internal consistency of the treatment processes. Figure 7 displays the results of the two linear regressions performed between total COD (DOC) and soluble COD (SCOD) for Cell 1 and Cell 2

Both treatment systems exhibited remarkably strong linear correlations between total and soluble chemical oxygen demand ($R^2=0.997$ for C1 and 0.983 for C2), demonstrating stable wastewater characteristics and confirming data consistency. The marginally higher R^2 value observed in the hydrochar-amended system indicates an additional stabilizing influence of the material.

Statistical Analysis and Significance

Normality tests (Shapiro-Wilk) indicated that none of the variables follow a normal distribution ($p<0.0001$ for all), justifying the use of nonparametric tests, such as the Mann-Whitney U test, for statistical comparisons.

The results of the statistical analysis reveal that, despite the numerical differences observed in removal efficiencies, these were not statistically significant for total COD ($p=0.1521$) or for soluble COD ($p=0.5818$). However, it is important to note that for soluble COD, a significant difference ($p=0.0025$) was found between the systems, suggesting that hydrochar might have a differential effect on the soluble fraction of chemical oxygen demand.

Implications for Hydrochar Application

The high standard deviation in the removal efficiencies of C1 (120.8% for total COD and 119.0% for soluble COD) indicates high variability in the system's performance with hydrochar, possibly due to irregular compound release from the material during the study period. This variability decreases considerably in the control system C2 (36.6% for total COD and 33.5% for soluble COD), suggesting more stable behavior in the absence of the amendment.

The results support the hypothesis that citrus waste hydrochar can improve the treatment of sugar cane industry wastewater, but implementation requires specific considerations. The prolonged stabilization period observed suggests the need to precondition the material before use in water treatment applications.

The recovery of system C1 after the initial period and its subsequent outperformance of the control system in the advanced phases suggest that, with optimizations in hydrochar pretreatment and extended operating periods, this material could offer significant advantages. Future studies should explore pre-stabilization methods that minimize initial leaching and accelerate the development of beneficial microbial communities.

Optimal hydrochar dosing, its interactions with different plant species in wetlands, and the monitoring of additional parameters, such as nutrients and heavy metals, constitute promising research lines to maximize the potential of this sustainable material in wastewater treatment applications.

The morphological development and growth dynamics of the plant species throughout the experimental period are detailed in Tables 3, 4, 5, and 6. Table 3 summarizes the analysis of stem elongation, including growth rates for individual specimens (SL_GRS1-3), the periods of highest and lowest growth (SL_HGP, SL_LGP), average variability (SL_AV), and a normality test (SL_SWT). Table 4 presents data on leaf production, detailing the growth rate per specimen (NLGRS1-3), leaf stability analysis (NLLSAS1-3), the overall rate trend (NLRT), and the total estimated change in leaf count (NLTEC). Furthermore, Table 5 provides a comprehensive overview of leaf length metrics, capturing the highest and lowest cumulative growth per specimen (LL_LHCG1-3, LL_LLCG1-3), key growth periods (LL_PHMR, LL_PLV), the overall growth range (LL_GRMIN, LL_GRMAX), and the average total growth (LL_AT). Finally, Table 6

Table 3. Stem growth. SL_GRS (Stem Length Grow Rate Specimen 1 2 3), SL_HGP (Stem Length Highest Growth period), SL_LGP (Stem Length Lowest Growth Period), SL_AV (Stem Length Average Variability), SL_SWT (Stem Length Shapiro Wilk Test).

Specimen	SL_GRS1 (%)	SL_GRS2 (%)	SL_GRS3 (%)	SL_HGP (%)	SL_LGP (%)	SL_SHG (%)	SL_AV (%)	SL_SWT
C1_Typha Latifolia	100	120	71.43	44.76	3.03	120	76.04	0.0002
C2_Typha Latifolia	83.33	71.43	100	23.61	7.04	100	41.4	0.0089
C1_Spathiphy Wallisii	171.43	166.67	150	71.43	53.49	171.43	12.1	0.4052
C2_Spathiphy Wallisii	120	133.33	116.67	51.11	47.93	133.33	10.88	0.6968
C1_Canna Indica	195.24	183.33	165.38	68.89	21.39	195.24	14.01	0.2076
C2_Canna Indica	155	126.09	115.38	45.26	24.07	155	11.61	0.3359

Table 4. Leaf count. NLGRS (Number of Leves Grow Rate Specimen), NLLSAS (Number of Leves Leaf Stability Analysis Specimen 1), NLRT (Number of Leves Rate Trend), NLTEC (Number of Leves Total Estimated Change).

Specimen	NL_GRS1 (%)	NL_GRS2 (%)	NL_GRS3 (%)	NL_LSAS1 (%)	NL_LSAS2 (%)	NL_LSAS3 (%)	NL_RT (%)	NL_TEC (%)
C1_Typha L.	50	50	100	1.12	0.5	1.37	−3.25	−16.27
C2_Typha L.	100	200	25	1.07	1.49	0.96	−1.28	−6.39
C1_Spathiphy W.	125	50	60	2.05	0.94	1.41	−52.14	−104.29
C2_Spathiphy W.	25	60	50	0.47	1.25	0.94	−33.57	−67.14
C1_Canna I.	0	50	75	1	0.74	1.12	−13.65	−40.95
C2_Canna I.	155	126	115.38	−8.31	−24.92		−8.31	−24.92

Table 5. Leaf length. LL_LHCG (Leaves length highest cumulative growth), LL_LLCG1 (Leaves lowest cumulative growth), LL_PHMR (Leaves Lowest Period with Highest mean rate), LL_PLV (Leaves Lowest Period with lowest variability), LL_GRMIN (Leaves Lowest Overall growth range min), LL_GRMAX (Leaves Lowest Overall growth range max), LL_AT (Leaves Lowest Average total growth).

Specimen	LL_ LHCG1 (%)	LL_ LHCG2 (%)	LL_ LHCG3 (%)	LL_ LLCG1 (%)	LL_ LLCG2 (%)	LL_ LLCG3 (%)	LL_ PHMR (%)	LL_PLV (%)	LL_ GRMIN (%)	LL_ GRMAX (%)	LL_AT (%)
C1_Typha L.	1680	1660	1416.67	955.56	955.56	1185.71	18.75	260	18.75	260	1293.36
C2_Typha L.	1320	914.29	912.5	844.44	887.5	912.5	217.14	17.54	18.33	260	978.75
C1_Spathiphy W.	220	220	183.33	81.82	90.91	111.11	70.97	33.66	31.25	150	146.28
C2_Spathiphy W.	275	266.67	242.86	109.09	125	125	78.9	42.56	31.25	133.33	183.6
C2_Canna I.	235	204.76	190	132	132	134.78	55.05	22.97	14.81	127.27	166.67
C2_Canna I.	266.67	220	211.11	136.36	161.54	181.82	61.21	20.23	15.62	114.29	193.34

Table 6. Leaf Width. WL_FAW (Wide Leaves Final Average Witdh), WL_FSD (Wide Leaves Final Standard Deviation), WL_FCV (Wide Leaves Final Coefficient Variation), WL_PGE (Wide Leaves Period Greatest Expansion), WL_PLE(Wide Leaves Period Least Expansion), WL_LGE (Wide Leaves Leaf Greatest Expansion), WL_LLE (Wide Leaves Leaf Least expansion), WL_FSD (Wide Leaves Final Stability), WL_DC (Wide Leaves Data Completeness).

Specimen	WL_FAW (%)	WL_FSD (%)	WL_FCV (%)	WL_PGE (%)	WL_PLE (%)	WL_LGE (%)	WL_ LLE (%)	WL_FS (%)	WL_DC (%)
C1_Typha L.	1.933	0.249	12.9	24.07	0	100	33.33	0.249	74.5
C2_Typha L.	1.933	0.17	8.79	27.78	3.03	33.33	0	0.17	62
C1_Spathiphy W.	8.435	2.356	31.69	45.66	45.21	200	0	2.356	82.6
C2_Spathiphy W.	8.33	2.357	28.28	41.8	31.58	133.33	12.5	2.357	82.5
C2_Canna I.	12.833	3.096	24.12	36.1	17.17	114.29	55.56	3.096	84.2
C2_Canna I.	10.667	2.385	22.36	41.72	21.35	160	37.5	2.385	83.8

compiles the results for leaf width, reporting the final average width and its dispersion (WL_FAW, WL_FSD, WL_FCV), the periods and specific leaves of most significant and least expansion (WL_PGE, WL_PLE, WL_LGE, WL_LLE), along with final stability and data completeness (WL_FSD, WL_DC).

Monitoring and Evaluation of Plant Development

The phytoremediation performance was assessed through comprehensive growth measurements of three plant species: *Canna indica*, *Spathiphyllum wallisii*, and *Typha latifolia*. The experimental setup consisted of two parallel constructed wetlands: C1 (amended with citrus waste hydrochar) and C2 (control system without amendment). Each wetland contained three specimens per species, totaling nine plants per system and eighteen plants across the entire experiment.

Four key morphological parameters were monitored periodically throughout the study period:

a) Stem growth (vertical development)
b) Leaf count (number of leaves per plant)
c) Leaf length (from base to apex)
d) Leaf width (maximum blade width)

This replicated design ($n=3$ per species per treatment) enabled robust statistical comparisons between the hydrochar-amended and control systems while accounting for intraspecies variability.

Statistically Significant Results

The statistical analysis demonstrated that the hydrochar amendment induced species-specific responses, with both notable improvements and particular trade-offs in growth parameters. Figure 8 shows the analysis of species performance based on four metrics: (A) maximum stem growth, (B) average leaf count, (C) average leaf length, and (D) average leaf width.

The analysis identified four key metrics where hydrochar amendment produced statistically significant effects ($p<0.05$):

a) *Canna indica* exhibited a 37.2% improvement in stem length ($p=0.0286$) but a substantial 68.5% reduction in leaf count ($p=0.0225$).
b) *Spathiphyllum wallisii* showed a 31.9% enhancement in stem length ($p=0.0088$).
c) *Typha latifolia* demonstrated an exceptional 142.0% increase in leaf length ($p=0.0124$).

Species-Specific Response Patterns

Canna indica displayed a mixed response to hydrochar amendment, characterized by a clear trade-off between structural growth and foliar production. The amendment promoted a +37.2% improvement of stem length and +20.3% enhancement of leaf width. However, these gains were accompanied by a −68.5% reduction of leaf count and a −9.7% decrease of leaf length. This pattern suggests that Canna Indica reallocated resources toward structural reinforcement and leaf thickening at the expense of foliar expansion and production. In contrast to *Canna indica*, *Spathiphyllum wallisii* responded uniformly positively to hydrochar amendment, showing enhancements across all measured parameters of 31.9%

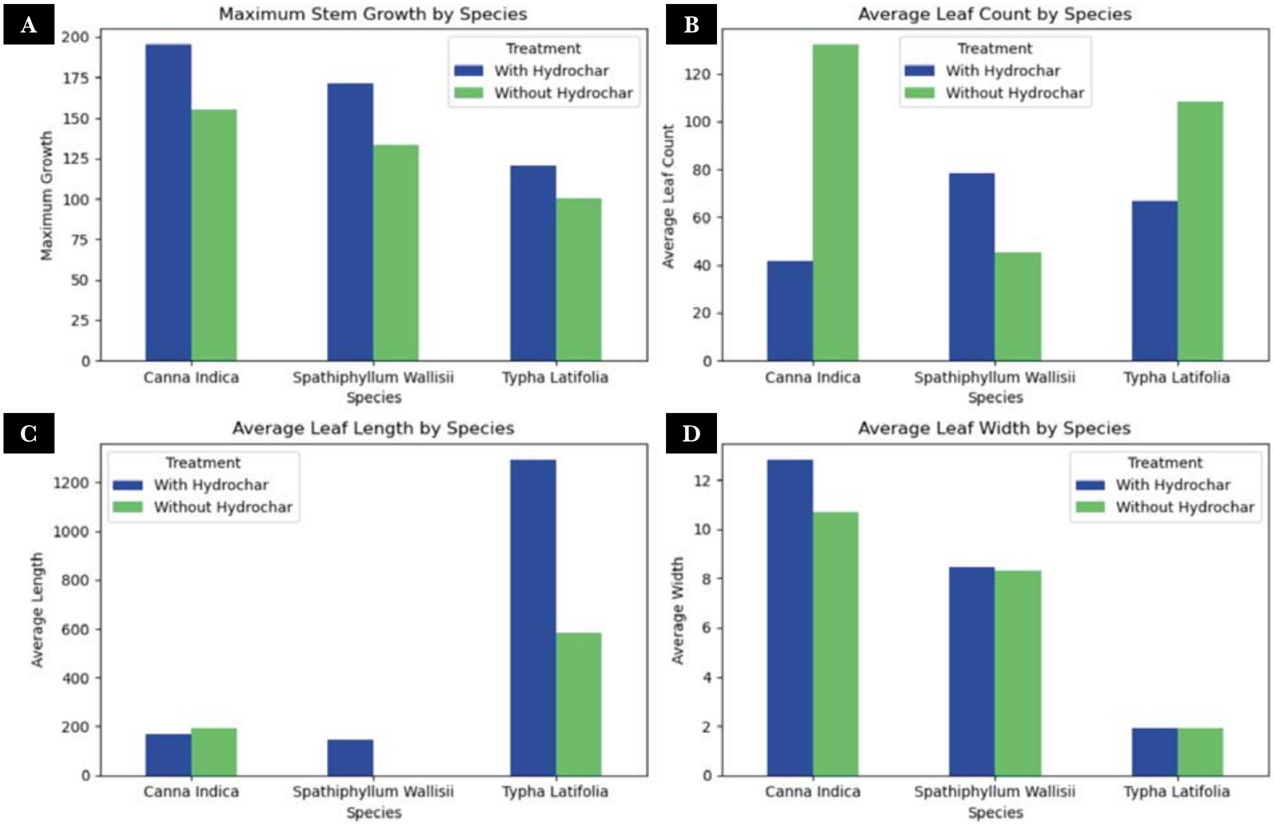


Figure 8. Species Responses. A) Maximum Stem Growth by Species, B) Average Leaf Count by Species, C) Average Leaf Length by Species, D) Average Leaf Width by Species.

improvement of stem length, +74.1% increase of leaf count, and +1.3% enhancement of leaf width. This consistent positive response indicates that *Spathiphyllum wallisii* effectively utilized the improved growing conditions provided by the hydrochar without apparent resource allocation conflicts. *Typha latifolia* exhibited the most dramatic response to hydrochar amendment, characterized by an extreme specialization in leaf development with +142% remarkable improvement of leaf length, +14.4 moderate enhancement of stem length, and −38.5% reduction of leaf count. This response pattern indicates a strategy of resource optimization, in which the species prioritized the development of exceptionally long leaves over maintaining foliar density. Figure 9 shows a heat map analyzing the effect of hydrochar on plant development. The rows represent the different species, while the columns correspond to the key morphological parameters: leaf count, leaf length, leaf width, and stem length.

Discussion of Growth Patterns

The differential responses observed among the three species highlight distinct physiological strategies for adapting to the hydrochar-amended environment. The trade-offs observed in *Canna indica* and *Typha latifolia* suggest that hydrochar may influence hormonal balances or resource allocation priorities, leading to specialized growth patterns rather than generalized improvement.

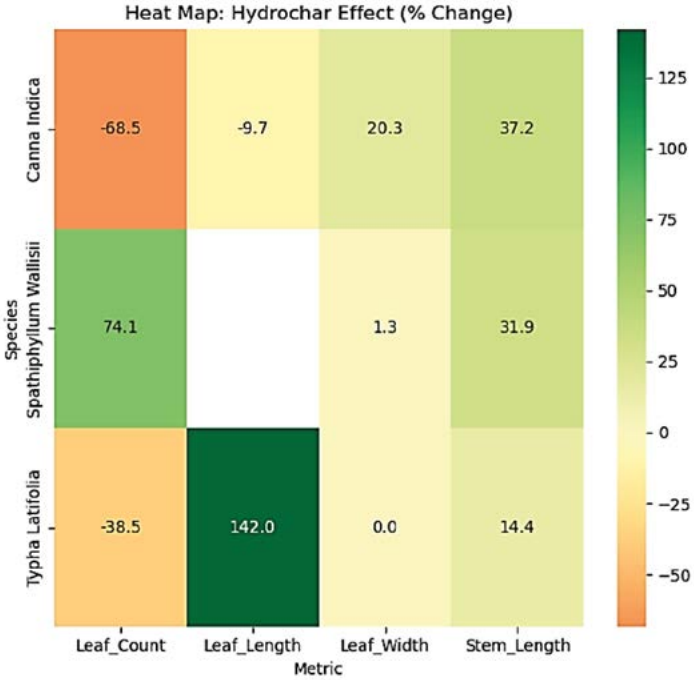


Figure 9. Heat Map Hidrochar Effect (% change).

The exceptional performance of *Typha latifolia* in leaf length development (+142.0%) positions it as a particularly suitable species for hydrochar-amended constructed wetlands, where maximum vertical growth and canopy development are desired objectives.

The consistent positive response of *Spathiphyllum wallisii* across all metrics makes it a reliable choice for general phytoremediation applications where balanced growth is preferred. The results demonstrate that citrus waste hydrochar significantly influences plant development in constructed wetlands. However, its effects are highly species-specific, resulting in distinct growth strategies that must be considered when selecting plant species for specific treatment objectives.

Overall Performance Assessment

Figure 10 illustrates the percentage change in the measured morphological parameters induced by the hydrochar treatment. The y-axis lists the twelve variables, comprising leaf width, leaf length, leaf count, and stem length for each of the three studied species, allowing for a direct comparison of the treatment's impact on individual growth metrics. The comprehensive analysis of plant growth parameters revealed a strongly positive net effect of citrus waste hydrochar amendment in the constructed wetland system. Of the 11 metrics evaluated across three plant species, seven improved with hydrochar amendment, three decreased, and one remained unchanged. This 7:3 improvement ratio demonstrates a clearly beneficial effect of hydrochar on plant development in constructed wetlands.

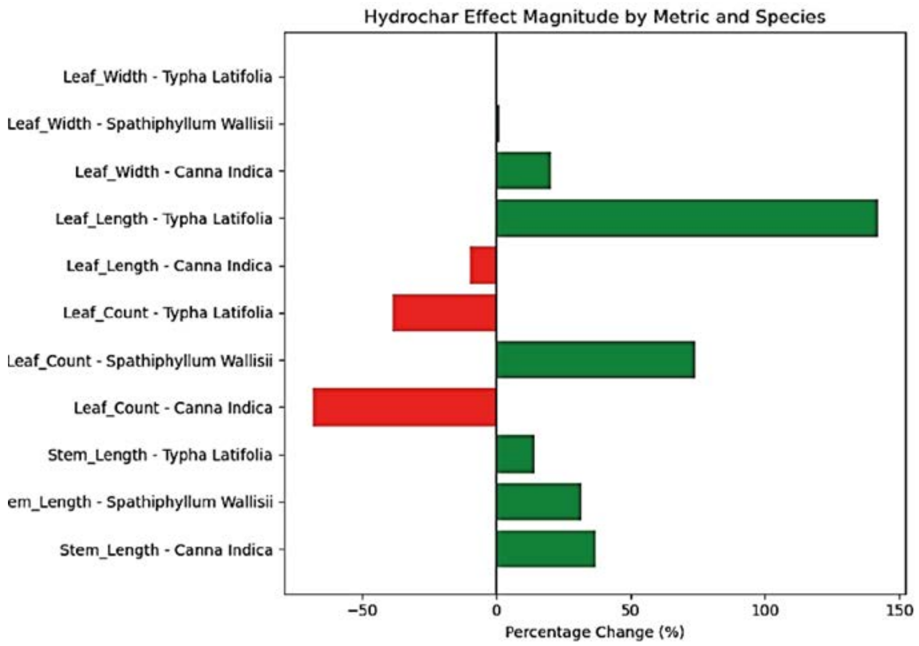


Figure 10. Hydrochar effect magnitude by Metric and Species.

Species Performance Ranking

Based on the comprehensive metric analysis, *Typha latifolia* emerged as the species that benefited most from hydrochar amendment. This conclusion is supported by its extraordinary performance in leaf length development (+142.0%), combined with moderate improvements in stem length (+14.4%) and stable leaf width maintenance. The species demonstrated an efficient growth strategy that prioritized the development of extensive leaf structures, which is particularly valuable for wastewater treatment applications where maximum surface area for microbial attachment and phytoremediation processes is desired.

The results demonstrate that citrus waste hydrochar serves as an effective growth enhancer in constructed wetlands, with the specific response patterns varying according to species-specific physiological characteristics and resource allocation strategies. Representative photographs of the two plant species used in the study, *Typha latifolia* and *Canna indica*, are shown in Figure 11(A) and 11(B), respectively.

CONCLUSIONS

This study demonstrates that citrus waste hydrochar represents a valuable soil amendment for constructed wetlands treating sugarcane industry wastewater. However, its application requires careful consideration of temporal dynamics and species-specific responses. The main conclusions derived from the experimental work are:

1. Hydrochar amendment significantly enhances long-term treatment performance, with the experimental system (C1) achieving 69.3% COD removal during the maturation phase compared to 65.5% in the control system, despite initial leaching

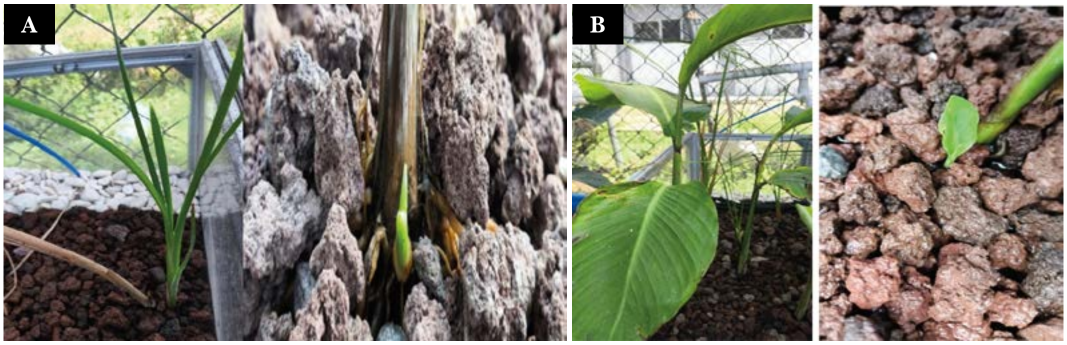


Figure 11. Plant species. A) *Typha latifolia* and B) *Canna indica*.

phenomena that caused negative removal efficiencies during the first 30 days of operation.

2. The strong linear correlation between total and soluble COD (R^2=0.997 in C1, R^2=0.983 in C2) confirms wastewater composition stability. It validates the internal consistency of the treatment processes, with hydrochar providing an additional stabilizing effect on the system.
3. Plant responses to hydrochar amendment were highly species-specific. *Typha latifolia* exhibited the most pronounced positive response with 142.0% greater leaf length development, making it particularly suitable for hydrochar-amended systems where maximum phytoremediation surface area is desired.
4. *Spathiphyllum wallisii* showed consistent improvement across all growth parameters (+31.9% stem length, +74.1% leaf count), indicating balanced enhancement without resource allocation trade-offs. At the same time, *Canna indica* displayed mixed responses with structural improvements at the expense of foliar production.
5. The integration of citrus waste hydrochar aligns with circular economy principles, transforming agricultural residues into valuable amendments that enhance both treatment efficiency and plant development in constructed wetlands, providing a sustainable alternative for sugarcane wastewater management.
6. Future applications should incorporate hydrochar pre-conditioning protocols to minimize initial leaching effects and consider species selection based on specific treatment objectives, with *Typha latifolia* recommended for maximum vertical development and *Spathiphyllum wallisii* for balanced growth performance.

ACKNOWLEDGMENTS

To the National Technological Institute of Mexico (TecNM) and the Secretariat of Education, Science, Technology, and Innovation (SECIHTI) for their institutional support and the resources provided for the development of this research project.

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