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EXPERIMENTAL EVIDENCE DRIVING INNOVATION IN NATURE-BASED SOLUTIONS FOR EMERGING CONTAMINANT REMOVAL

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Abstract

This study proposes a framework to innovate Nature-Based Solutions through substrate optimization for contaminants of emerging concern adsorption removal. Reactive materials were evaluated using physicochemical characterization, adsorption tests, lab-scale constructed wetlands, and pilot-scale validation. Sewage sludge biochar and coco coir showed the best removal performance. Systems amended with these substrates outperformed gravel controls under different water matrices, while pilot-scale tests confirmed the feasibility of biochar-based polishing under real conditions. Overall, the framework proved effective for identifying and validating high-performance substrates for advanced NbS applications.

Palabras Clave: constructed wetlands, wastewater, substrates.

1 Introduction

Nature-Based Solutions (NbS) have evolved from passive landscape elements into engineered systems for circular water management and climate-resilient infrastructure. In this transition, their role has expanded from mainly ecological functions to integrated systems that combine natural processes with engineered components [1]. By coupling physical, chemical, and biological mechanisms, NbS can achieve effective water treatment across a wide range of water types, while maintaining low energy demand and reduced operational costs compared to conventional treatment technologies [2].

However, the growing occurrence of contaminants of emerging concern (CECs), including pharmaceuticals, personal care products, pesticides, and endocrine-disrupting compounds, exposes a critical limitation in current NbS design [3]. These micropollutants are consistently detected at trace concentrations in wastewater effluents, surface waters, and stormwater-influenced systems. Their persistence is driven by structural stability and partial resistance to biodegradation, resulting in incomplete removal within standard NbS configurations [4].

To address this limitation and enhance the competitiveness of NbS compared to conventional treatment systems, this study proposes a systematic engineering framework for performance improvement through substrate optimization. Rather than treating material selection as an empirical or secondary design step, the framework redefines the filtration matrix as a controllable reactive interface, where contaminant removal can be intentionally tuned through targeted material selection and functional modification. In this way, NbS can be systematically upgraded to improve removal efficiency, broaden applicability to emerging contaminants, and strengthen their role as viable alternatives to conventional water treatment technologies.

In this context, enhancing NbS performance is approached through a structured framework that integrates:

(I) rational selection of candidate adsorbent materials and physicochemical characterization,
(II) performance-based ranking using adsorption experiments,
(III) laboratory-scale experimental validation, and
(IV) progressive upscaling from batch systems to continuous flow and pilot-scale validation.

This framework enables the identification of substrate configurations that maximize contaminant retention, extend effective residence time at the microscale, and promote synergistic interactions between adsorption, biodegradation, and plant-mediated processes.

2 Materials and methods

The experimental methodology follows a structured engineering framework for substrate optimization in NbS, progressing from material selection and characterization to batch adsorption testing, lab-scale validation in constructed wetlands, and pilot-scale demonstration under real operating conditions.

2.1 Substrate selection and characterization (Framework step I)

Based on an extensive screening of potential reactive materials for constructed wetlands and NbS applications, seven substrates were selected: chitosan, sewage sludge biochar (biochar SS), softwood pellet biochar (biochar SWP), coco coir, perlite, zeolites, and titanium dioxide (TiO_2). These materials were selected to represent a broad range of organic, mineral, and engineered adsorbent properties relevant for emerging contaminant removal. Chitosan (low molecular weight polymer) and TiO_2 (fine particulate material) were immobilized onto a zeolite support matrix.

All substrates were subjected to physicochemical and environmental characterization. Particle size distribution was determined using laser granulometry. Textural properties, including specific surface area and pore volume, and pore size distribution, were evaluated. Environmental safety was assessed through standardized leaching tests. The potential release of heavy metals (Ni, Pb, Cd, Cr, Cu, Hg, and Zn) was quantified in leachates using inductively coupled plasma mass spectrometry (ICP-MS).

2.2 Adsorption-based performance screening (Framework step II)

Batch adsorption experiments were conducted to evaluate the removal performance of the selected substrates. Both adsorption kinetics and equilibrium isotherms were assessed to determine uptake rates and maximum adsorption capacities. A synthetic contaminant mixture containing five representative CECs, diclofenac (DFC), azithromycin (AZI), imidacloprid (IMD), estrone (E1), and estradiol (E2), was prepared at an initial concentration of 400 µg/L.

Kinetic experiments were performed to evaluate time-dependent adsorption behavior. Equilibrium isotherms were conducted over a concentration range of 50–400 µg/L to determine adsorption capacity. Residual concentrations were quantified using liquid chromatography–tandem mass spectrometry (LC–MS/MS).

2.3 Lab-scale Nature-Based Solutions validation (Framework step III)

Based on adsorption performance and material characterization results, sewage sludge biochar (biochar SS) and coco coir were selected as the most promising substrates for integration into lab-scale vertical flow constructed wetlands (VF-CWs). The experimental design consisted of two configurations: (i) CWs systems filled with a mixture of biochar SS, coco coir, and gravel, and (ii) control systems containing only gravel.

The systems were operated continuously for four months under controlled hydraulic conditions. Three synthetic wastewater matrices were tested: municipal wastewater (WW), greywater (GW), and stormwater (SW), prepared following established formulations from previous studies. All matrices were spiked with the five-CEC. Influent and effluent samples were collected periodically throughout the experimental period. CEC concentrations were quantified using LC–MS/MS to evaluate removal performance under different water quality scenarios.

2.4 Pilot-scale validation under real conditions (Framework step IV)

Pilot-scale validation was conducted at the REFLET INRAE Research Platform (France) to evaluate system scalability and performance under real environmental conditions. An external downflow polishing column (internal diameter: 19.2 cm) was installed downstream of an aerated VF-CW treating real combined sewer overflow (CSO) effluent. The column contained a 20 cm reactive layer of biochar (0–4 mm particle size), sandwiched between glass bead layers to improve hydraulic distribution, minimize preferential flow, and reduce clogging risk.

The system was operated continuously for 78 days (September–November 2024) at a constant flow rate of 10 L h^{-1}, corresponding to an approximate hydraulic residence time of ~23 minutes. Influent and effluent samples were collected weekly, with increased sampling frequency during startup and final operational phases to capture breakthrough dynamics and transient behavior under real loading conditions.

3 Results and Discussion

3.1 Framework step I: Material characterization and screening

The initial application of the framework enabled a systematic discrimination of the seven tested substrates based on their physicochemical and environmental properties. Biochar SS exhibited the highest specific surface area, while coco coir showed the widest particle size distribution, providing a combination of micro- and macroporous structures. Most materials were predominantly mesoporous, although SWP biochar contained a higher micropore fraction. Perlite, zeolites, and TiO_2-based materials showed comparatively lower textural development.

Leaching tests confirmed environmental safety for all substrates, with heavy metal concentrations (Ni, Pb, Cd, Cr, Cu, Zn) remaining below regulatory thresholds. Overall, biochar SS and coco coir emerged as the most promising candidates based on their combined structural and environmental performance.

Overall, this first framework step established a clear physico-environmental hierarchy of materials, positioning biochar SS and coco coir as the most promising candidates for further performance evaluation.

3.2 Framework step II: Adsorption performance ranking

The second step of the framework translated material properties into functional performance through batch adsorption experiments. Biochar SS and coco coir were identified as the most effective substrates for CEC removal (Figure 1). The biochar exhibited consistently high and non-selective adsorption across all target compounds, whereas coco coir showed preferential affinity toward hormones and azithromycin. Lower performance was observed for biochar SWP, while negligible adsorption was recorded for perlite, zeolites, TiO_2-modified zeolites, and chitosan-based materials. This step was essential in refining the material pool, enabling the selection of SS biochar and coco coir as priority substrates for system-scale validation within the framework.

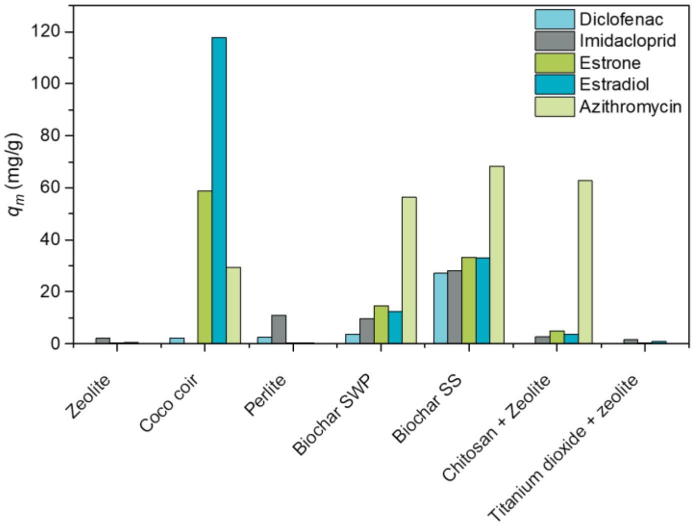

Figure 1. The maximum adsorption capacity of each material for the five contaminants.

3.3 Framework step III: Lab-scale Nature-Based Solutions performance

At lab scale, this step of the framework provided a critical validation of substrate performance under dynamic and realistic operational conditions, bridging the gap between batch adsorption screening and pilot-scale application. Its main relevance lies in confirming whether the performance gains identified in earlier stages translate into functional improvements at the system level.

Mixed substrates (SS biochar, coco coir, and gravel) consistently outperformed gravel-only controls across all water matrices, except for azithromycin in greywater. This confirms the ability of the framework to identify substrate configurations that enhance overall system performance beyond baseline conditions.

In synthetic wastewater, mixed systems achieved an average removal of 79% for diclofenac (with a maximum of 96%) and an average 93% for imidacloprid (with a maximum of 98%), while controls showed negligible performance (≤28% and ≤18% maximum, respectively) (Figure 2). Similar trends were observed in greywater and stormwater conditions, where mixed systems consistently outperformed controls, although azithromycin remained an outlier (Figures 3 and 4).

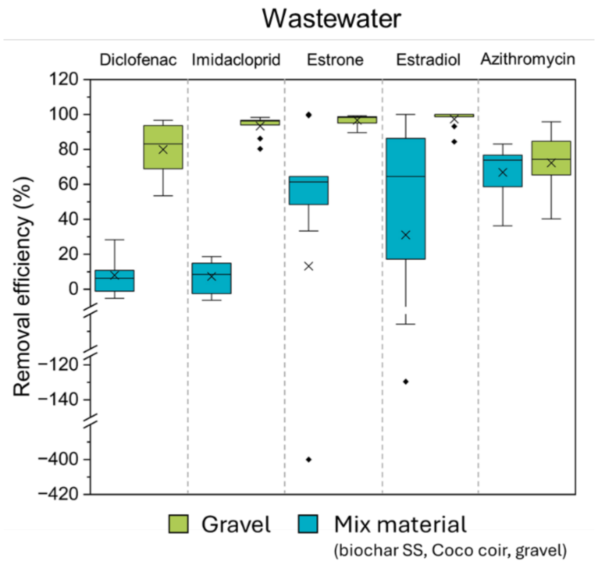

Figure 2. Average removal efficiency of emerging contaminants in lab-scale constructed wetlands with adsorbent materials fed by synthetic wastewater over four months.

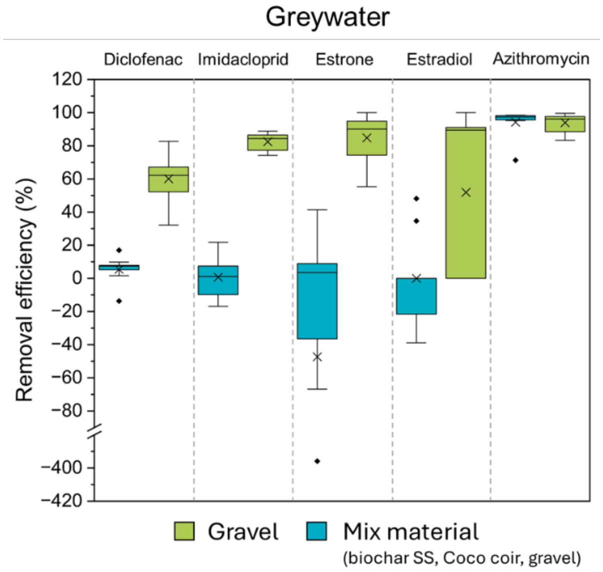

Figure 3. Average removal efficiency of emerging contaminants in lab-scale constructed wetlands with adsorbent materials fed by synthetic greywater over four months.

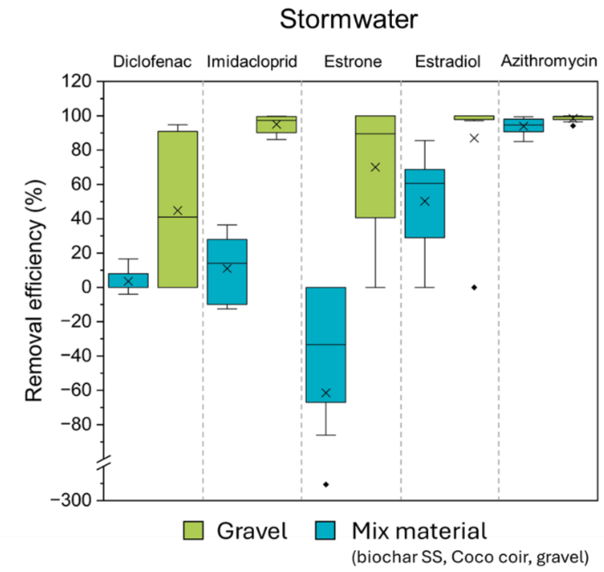

Figure 4. Average removal efficiency of emerging contaminants in lab-scale constructed wetlands with adsorbent materials fed by synthetic stormwater over four months.

Overall, this step was essential within the framework, as it validated that substrate-driven improvements persist under dynamic conditions and highlighted matrix- and compound-dependent limitations not captured in earlier stages.

3.4 Framework step IV: Pilot-scale validation

Pilot-scale results demonstrated the feasibility of using biochar as a polishing step under real conditions. The system showed stable retention for compounds such as benzotriazole, oxazepam, amisulpride, and irbesartan, with removal efficiencies up to ~30%. Irbesartan notably showed improved removal in the biochar column compared to the upstream VF-CW, highlighting its role as a complementary treatment stage. However, performance was compound-dependent: erythromycin and benzophenone showed poor retention, while DEET and diclofenac exhibited moderate and variable removal.

4 Conclusion

This study presents a structured experimental framework to innovate NbS through targeted substrate engineering for enhanced removal of CECs. By integrating material screening, adsorption-based ranking, lab-scale system validation, and pilot-scale implementation, the framework enables a systematic, evidence-based approach to transform substrate selection from an empirical practice into a performance-driven design strategy.

Across all stages, SS biochar and coco coir were consistently identified as the most effective substrates, confirming the robustness of the framework in linking material properties to treatment performance. Results demonstrate that adsorption-driven improvements observed at the material scale are effectively translated to dynamic and real operating conditions.

Lab- and pilot-scale validations further show that engineered substrates can significantly enhance NbS performance compared to conventional configurations, while also highlighting compound- and matrix-dependent limitations. Overall, this work contributes to the innovation of NbS by providing a transferable framework for the rational design of more efficient and resilient water treatment systems targeting emerging contaminants.

6 References

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