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Fish-Bone-Derived Porous Carbon/Hydroxyapatite Composite for Sustainable Treatment of Local Sludge Wastewater

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ABSTRACT

Fish-bone waste was valorized into a porous carbon/hydroxyapatite composite and evaluated for sustainable treatment of local sludge wastewater. Catfish bones collected in Sokoto State were washed, oven-dried, pulverized, impregnated with H_3PO_4 (1:3) for 24 h, carbonized at 350 °C for 30 min, neutralized, dried, and applied in a filtration column. The resulting material was characterized by FTIR, XRD, SEM, and EDX. FTIR revealed O–H, C–H, and oxygen-containing surface functionalities alongside prominent phosphate-associated bands, while XRD showed crystalline reflections consistent with retention of the calcium-phosphate mineral phase. SEM revealed a rough, porous carbonaceous matrix containing dispersed mineral domains, and EDX confirmed C, O, P, and Ca as the predominant elements, collectively supporting formation of a carbon/hydroxyapatite composite. Filtration markedly improved sludge-water quality, achieving 92.77% COD, 89.02% BOD_5, and 96.40% turbidity removal. The pH increased from 3.40 to 7.20, while dissolved oxygen increased from 0.56 to 6.20 mg/L. High removal efficiencies were also obtained for Cd (99.51%), Cr (92.93%), Cu (91.43%), and Zn (92.00%). These findings demonstrate the potential of fish-bone waste as a low-cost, sustainable precursor for producing a multifunctional carbon/mineral adsorbent for wastewater treatment and resource valorization.

KEYWORDS: fish bone; porous carbon; hydroxyapatite; sludge wastewater; heavy metals.

1. INTRODUCTION

Increasing pressure on freshwater resources, population growth, urbanization, and inadequate wastewater management have intensified the need for sustainable technologies that can improve wastewater quality while facilitating safe water reuse.[1,2] Wastewater and sludge-associated aqueous streams are chemically complex and may contain suspended and dissolved solids, biodegradable and oxidizable organic matter, nutrients, inorganic ions, and potentially toxic metals. The persistence of metal contaminants is of particular concern because they are non-biodegradable and may accumulate in environmental compartments if inadequately treated.[3–7] Consequently, the development of affordable treatment materials capable of removing multiple classes of contaminants while minimizing secondary waste and resource consumption remains an important objective of sustainable water management.

Among available treatment approaches, adsorption has received considerable attention because of its operational simplicity, flexibility, relatively low energy requirements, and suitability for both centralized and decentralized applications.[2,8–10] Recent developments have increasingly focused on porous and waste-derived adsorbents as alternatives to comparatively expensive conventional materials. Porous materials can provide large accessible surfaces and diverse adsorption sites for wastewater contaminants, while carbonaceous adsorbents offer tunable pore structures and surface functionalities that support interactions with both inorganic and organic species.[11,12] Biomass- and biowaste-derived carbon materials are particularly attractive from a sustainability perspective because their production can simultaneously valorize residual resources and generate functional materials for environmental remediation.[13–16]

Fish-processing residues represent a potentially valuable feedstock within this waste-to-resource framework. Fish bones contain an organic matrix together with a substantial calcium-phosphate mineral fraction, predominantly associated with hydroxyapatite (HAp). Unlike conventional lignocellulosic precursors used primarily to produce carbonaceous adsorbents, thermal processing of fish bone can therefore generate a heterogeneous carbon–mineral material in which residual carbonaceous structures coexist with HAp-rich inorganic phases.[17] Recent work has demonstrated that pyrolysis and activation of fish-bone biowaste can produce porous HAp-rich adsorbents containing residual carbon structures and surface functional groups suitable for aqueous contaminant removal.[18] This distinctive composition provides an opportunity to exploit fish-bone waste directly as a precursor for multifunctional adsorbents rather than removing its mineral fraction before carbonization.

Hydroxyapatite is particularly relevant to metal-contaminated wastewater because its calcium-phosphate framework exhibits strong affinity toward several metal ions through mechanisms that may include surface complexation, ion exchange, dissolution–precipitation, and incorporation into the mineral lattice. Accordingly, HAp-based materials have emerged as promising low-cost adsorbents for heavy metals and other aqueous pollutants.[19,20] Fish-bone-derived HAp has demonstrated effective Cd(II) removal from aqueous systems and real industrial wastewater[20], while recent investigations have further demonstrated substantial adsorption of Cr^{3+}, Ni^{2+}, and Zn^{2+} by HAp derived from different fish species, with removal performance strongly influenced by fish species, thermal treatment, and solution pH.[21] Waste-derived HAp can therefore contribute simultaneously to contaminant immobilization and the productive utilization of calcium-phosphate-rich biological residues.

Combining a porous carbonaceous phase with naturally occurring HAp may offer additional advantages over either component considered independently. The carbonaceous fraction can contribute porosity, accessible surface area, and oxygen-containing functional groups, whereas the calcium-phosphate phase can provide chemically active sites with strong affinity for metal ions. Such complementary characteristics may broaden the range of interactions available for contaminant sequestration. The feasibility of this composite approach is supported by studies demonstrating that waste-derived HAp-containing composites can exhibit high porosity and substantial adsorption capacities for metal ions and other wastewater contaminants.[22] More broadly, the integration of waste-derived carbon materials with functional inorganic phases is consistent with current efforts to develop multifunctional adsorbents that couple high treatment performance with resource recovery and circular-economy principles.

Against this background, the present study investigates a fish-bone-derived porous carbon/hydroxyapatite composite for sustainable treatment of local sludge wastewater. By converting locally available fish-bone waste into a functional carbon–mineral adsorbent, the study explores an approach that integrates waste valorization, contaminant removal, and sustainable wastewater management.

2. MATERIALS AND METHODS

2.1 Preparation of fish-bone adsorbent

Catfish bones were separated from residual tissue, washed with distilled water and oven-dried at 150 °C for 24 h. The dried material was crushed and sieved to approximately 110 mesh. Fish-bone powder was impregnated with 75% H_3PO_4 at a solid-to-acid ratio of 1:3 for 24 h, filtered, dried and carbonized in a furnace at 350 °C for 30 min. After cooling, the product was washed with NaOH solution and deionized water until the washings reached approximately neutral pH. The material was then dried and pulverized before use. This preparation procedure is consistent with established approaches involving acid modification and thermal conversion of fish-bone waste into carbonaceous/HAp-containing adsorbent materials[18,23].

2.2 Wastewater collection and filtration

Sludge wastewater was collected from Madorawa, Bodinga Local Government Area, Sokoto State. Large particulate matter was removed before treatment. The prepared fish-bone material was used in a burette-column filtration arrangement in which cotton supported the adsorbent bed. Wastewater was contacted with the adsorbent and passed through the column, and the filtrate was collected for analysis as described elsewhere.[16]

2.3 Characterization and water-quality analyses

FTIR spectra were recorded over 4000–400 cm^{-1} to examine surface functional groups. XRD was used to assess crystalline phases and structural order; the study adopted a typical 2θ range of approximately 10–80° using Cu Kα radiation (λ = 1.5406 Å). SEM was used to examine morphology, while EDX associated with the SEM analysis was used to assess elemental composition. Physicochemical parameters were determined before and after treatment using standard methods.[24] Heavy-metal concentrations of Cd, Cr, Cu and Zn were determined by atomic absorption spectroscopy (AAS). Removal efficiency was calculated as $R\ (\%) = [(C_i - C_f)/C_i] \times 100$, where C_i and C_f are the concentrations before and after filtration, respectively. For dissolved oxygen, the direction of improvement was reported as an increase rather than removal.

3. RESULTS AND DISCUSSION

3.1 Structural and surface characterization of the Fish-Bone-Derived Porous Carbon/Hydroxyapatite Composite

The FTIR spectrum of the fish-bone-derived material (Figure 1a) exhibits a weak, broad absorption in the hydroxyl stretching region at approximately 3370–3540 cm^{-1}, together with a C–H stretching feature around 2875 cm^{-1}. The most prominent absorptions occur below approximately 1400 cm^{-1}, particularly within the 1200–900 cm^{-1} fingerprint region. Rather than being attributed exclusively to carbonaceous functionalities, these bands are more appropriately interpreted as contributions from overlapping P–O vibrations of the retained calcium-phosphate phase and C–O-containing surface groups of the carbonaceous matrix. This interpretation is consistent with recent studies of fish-bone-derived hydroxyapatite (HAp), in which characteristic phosphate stretching vibrations have been reported at approximately 1000–1080 cm^{-1}, together with hydroxyl and residual organic functionalities.[21] Similarly, HAp extracted from fish bone has shown characteristic phosphate bands at approximately 1044 and 963 cm^{-1}.[25] The FTIR spectrum therefore indicates the coexistence of surface-functionalized carbonaceous matter and a retained phosphate-rich mineral phase, supporting assignment of the material as a carbon/HAp composite rather than phase-pure porous carbon.

The XRD pattern (Figure 1b) contains several relatively sharp diffraction reflections superimposed on a broader background, indicating the coexistence of crystalline and comparatively disordered components. Such a pattern is inconsistent with assignment of the material as exclusively amorphous carbon and instead demonstrates retention of a crystalline inorganic phase following carbonization at 350 °C. This interpretation agrees with recent characterization of fish-bone-derived HAp, for which well-defined XRD reflections characteristic of crystalline HAp have been observed.[25] More importantly, Zhang et al. (2026) recently demonstrated that pyrolysis and activation of fish bone can yield an HAp-rich adsorbent containing a residual carbon structure, providing direct precedent for the coexistence of carbonaceous and calcium-phosphate phases in thermally processed fish-bone materials.[18] When considered alongside the phosphate-associated FTIR bands and the Ca- and P-rich EDX response, the present XRD pattern therefore strongly supports identification of the material as a porous carbon/hydroxyapatite composite.

The SEM micrograph (Figure 1c) reveals an irregular, rough and highly textured morphology characterized by interconnected cavities and agglomerated domains. The porous matrix is interspersed with angular, comparatively compact particles that are morphologically distinct from the surrounding carbonaceous structure. In conjunction with the FTIR and XRD results, these features are consistent with HAp-rich mineral domains retained within and on the surface of a porous carbonaceous matrix. Comparable heterogeneous and porous morphologies have been observed in fish-bone-derived HAp materials, while porosity has been associated with improved accessibility of aqueous contaminants to adsorption sites. Recent work on thermally treated fish bone likewise demonstrates that pyrolysis and activation can improve the porosity and surface functionality of HAp-rich fish-bone adsorbents.[18] The interconnected cavities observed here may consequently facilitate access of dissolved species to external and internal adsorption sites and reduce diffusion distances within the adsorbent. Nevertheless, the SEM image provides morphological rather than quantitative porosity information; parameters such as specific surface area, pore volume and pore-size distribution would require complementary gas-adsorption measurements.

The EDX spectrum and elemental maps (Figure 1d) provide further evidence for the composite nature of the material. The analysis is dominated by C, O, P and Ca, with smaller contributions from Na and Si. The strong carbon signal confirms retention of a substantial carbonaceous component, whereas the simultaneous occurrence of Ca, P and O is consistent with the calcium-phosphate/HAp phase identified from FTIR and XRD. Fish-bone-derived HAp is similarly characterized by Ca- and P-rich compositions, and recent studies routinely combine SEM–EDX with FTIR and XRD to establish the chemical and structural characteristics of such materials.[21] The elemental maps additionally show that C, O, P and Ca are distributed throughout the examined region, although local variations are evident, consistent with a heterogeneous carbon–mineral architecture. The EDX-derived Ca/P atomic ratio of approximately 1.34 is lower than the theoretical value of 1.67 for stoichiometric hydroxyapatite and should therefore not be interpreted as evidence of phase-pure stoichiometric HAp. Rather, it may reflect a calcium-deficient or compositionally heterogeneous phosphate-containing phase, contributions from other Ca- or P-containing species, and the local and semi-quantitative nature of EDX analysis. Accordingly, EDX is used primarily here to establish elemental identity and spatial association rather than precise bulk stoichiometry.

Together, the FTIR, XRD, SEM and EDX results provide mutually reinforcing evidence that carbonization of fish bone at 350 °C preserved a substantial fraction of the native calcium-phosphate mineral while converting part of the organic component into a porous carbonaceous matrix. The resulting material is therefore more appropriately described as a fish-bone-derived porous carbon/hydroxyapatite composite than as phase-pure activated or nanoporous carbon. This dual-phase architecture is potentially advantageous for wastewater treatment because the carbonaceous component can contribute accessible pores and surface functional groups, while HAp provides chemically active phosphate- and calcium-associated sites capable of interacting with metal ions. HAp-based materials have been reported to remove metals through combinations of surface complexation, ion exchange and dissolution–precipitation processes, depending on the adsorbate and solution conditions. Fish-bone-derived HAp has, for example, demonstrated effective Cd^{2+} removal from real wastewater, supporting its potential as a waste-derived adsorbent for metal-contaminated aqueous systems. More recent studies further demonstrate the capacity of fish-bone-derived HAp and HAp-containing composites to remove multiple metal ions from water and wastewater.

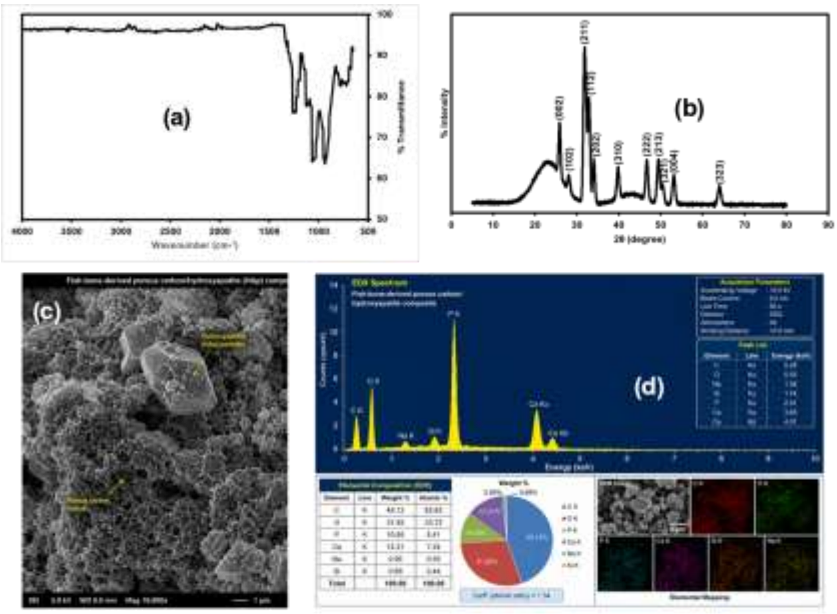

Figure 1. Characterization of the fish-bone-derived porous carbon/hydroxyapatite (HAp) composite: **(a)** FTIR spectrum showing characteristic surface functional groups; **(b)** XRD pattern confirming the crystalline phases of the composite; **(c)** SEM micrograph showing the porous carbon matrix and embedded HAp-rich particles; and **(d)** EDX spectrum, elemental composition, and elemental mapping confirming the presence and distribution of C, O, P, Ca, Na, and Si.

3.2 Physicochemical performance of composite

Filtration with the fish-bone-derived porous carbon/hydroxyapatite composite substantially improved the physicochemical characteristics of the sludge water (Table 1). The initially acidic pH increased from 3.40 to 7.20, bringing the treated water within the stated acceptable range of 6.5–8.5. This shift is important because pH influences contaminant speciation, adsorption behaviour and overall treatment performance. WHO similarly recognizes pH as an important operational water-quality parameter.[26]

Electrical conductivity (EC) decreased from 940 to 245 µS/cm (73.94%), while total dissolved solids (TDS) declined from 604 to 198 mg/L (67.22%), indicating substantial reduction of dissolved ionic constituents. The final values were below the NSDWQ limits reported in Table 1. WHO also recognizes TDS as an important indicator of drinking-water acceptability, although no health-based guideline value is established for TDS.[27]

Marked reductions were observed in oxygen-demanding and particulate pollutants. COD decreased from 249 to 18.0 mg/L, corresponding to 92.77% removal, while BOD_5 declined from 41.0 to 4.50 mg/L (89.02%). Turbidity exhibited the highest removal efficiency, decreasing from 75.0 to 2.70 NTU (96.40%). Effective turbidity reduction is particularly important because suspended and colloidal particles can impair water quality and interfere with subsequent treatment and disinfection processes[26,27]

Concurrently, dissolved oxygen increased from 0.56 to 6.20 mg/L, consistent with the substantial reduction in oxygen-demanding constituents. Overall, the simultaneous improvements in pH, EC, TDS, COD, BOD_5, turbidity and DO demonstrate effective attenuation of dissolved, particulate and organic pollutant loads. These results indicate strong treatment performance of the fish-bone-derived composite, although compliance with these physicochemical parameters alone should not be interpreted as confirmation of drinking-water safety without complementary microbiological and contaminant-specific assessments.[26]

Table 1. Physicochemical characteristics of sludge water before and after filtration.

Parameter	Before filtration	After filtration	Change/Removal (%)	WHO/NSDWQ guideline/limit[28,29]	Assessment after filtration
pH	3.40	7.20	—	6.5–8.5	Within acceptable range
EC (µS/cm)	940	245	73.94	1,000 µS/cm†	Within NSDWQ limit
TDS (mg/L)	604	198	67.22	500 mg/L†	Within NSDWQ limit
DO (mg/L)	0.56	6.20	+1007.14‡	-	-
COD (mg/L)	249	18.0	92.77	-	-
BOD_5 (mg/L)	41.0	4.50	89.02	-	-
Turbidity (NTU)	75.0	2.70	96.40	≤5 NTU	Within commonly applied drinking-water limit

† Nigerian Standard for Drinking Water Quality (NSDWQ), NIS 554:2015, Standards Organisation of Nigeria (SON).[28]

3.3 Heavy-Metal Removal

The fish-bone-derived porous carbon/hydroxyapatite composite demonstrated high removal efficiencies for all four target metals (Table 2). Cd decreased from 0.570 to 0.0028 mg/L, corresponding to 99.51% removal, and the residual concentration was within the WHO guideline value of 0.003 mg/L.

Cr decreased from 0.580 to 0.0410 mg/L (92.93% removal), also below the WHO guideline of 0.05 mg/L, while Cu declined from 0.910 to 0.0780 mg/L (91.43% removal), substantially below its 2.0 mg/L guideline value. Zn decreased from 3.500 to 0.2800 mg/L (92.00% removal); WHO does not establish a health-based guideline value for Zn at concentrations normally encountered in drinking water.[26,27]

The consistently high removal (>91%) is compatible with the complementary surface chemistry of the carbon/HAp composite. Oxygen-containing carbon functionalities may contribute through surface complexation and electrostatic interactions, while hydroxyapatite can immobilize metal ions through ion exchange, surface complexation and dissolution–precipitation processes, as reported for waste-derived HAp adsorbents.[20] However, adsorption kinetics, isotherms and post-adsorption characterization would be required to establish the dominant removal mechanisms quantitatively.

Table 2. Concentrations and removal efficiencies of selected heavy metals in sludge water before and after filtration.

Heavy metal	Before filtration (mg/L)	After filtration (mg/L)	Removal (%)	WHO guideline (mg/L)[26,27]	Assessment after filtration
Cd	0.570	0.0028	99.51	0.003	Within WHO guideline
Cr	0.580	0.0410	92.93	0.05	Within WHO guideline
Cu	0.910	0.0780	91.43	2.0	Within WHO guideline
Zn	3.500	0.2800	92.00	No WHO health-based given value	Substantially reduced

4. CONCLUSION

A porous carbon/hydroxyapatite composite was prepared from waste catfish bones through H_3PO_4 impregnation followed by carbonization at 350 °C. FTIR, XRD, SEM and EDX analyses collectively confirmed a porous carbonaceous matrix containing retained calcium-phosphate/hydroxyapatite phases. The composite markedly improved sludge-water quality, achieving 92.77% COD, 89.02% BOD_5 and 96.40% turbidity removal, accompanied by a substantial increase in dissolved oxygen. High removal efficiencies were also obtained for the target heavy metals, reaching 99.51% for Cd, 92.93% for Cr, 91.43% for Cu and 92.00% for Zn. These findings demonstrate the potential of fish-bone waste as a low-cost and sustainable precursor for producing a multifunctional carbon/mineral adsorbent for wastewater treatment. Further studies should incorporate BET surface-area and pore-size analyses, calibrated XRD phase identification, adsorption kinetics and isotherms, regeneration and reuse studies, and post-adsorption characterization to establish adsorption mechanisms, long-term performance and practical applicability before large-scale deployment or claims regarding drinking-water safety are considered.

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