Received: 20 February 2023

Revised: 25 April 2023

(wileyonlinelibrary.com) DOI 10.1002/jctb.7415

A review on the versatility of *Carica papaya* seed: an agrogenic waste for the removal of organic, inorganic and microbial contaminants in water

Ajibola A Bayode,^{a*} [©] Eric T Anthony,^b Odunayo T Ore,^c Moses O Alfred,^{a,d} Daniel T Koko,^{a,d} Emmanuel I Unuabonah,^{a,d} Brigitte Helmreich^e and Martins O Omorogie^{a,d,e*} [©]

Abstract

The advent of civilization, coupled with growing industrialization in many countries, is placing more demand on the available water sources. At the same time, the daily surge in wastes generated by man's anthropogenic activities has led to microbial, organic and inorganic contamination of water sources. Based on available evidence, significant research efforts are being made into the use of low-cost agricultural materials such as *Carica papaya* seed (CPS) in the removal of these contaminants from water sources in a bid to provide clean water. In the present review, the organic, inorganic and microbial contaminants in waters were elucidated. Furthermore, the chemical composition of the CPS was illustrated. The adsorption capacity and efficiency of CPS and their composites in the remediation of the selected contaminants were discussed while identifying the various factors affecting the adsorption efficiency. Finally, the reusability of this agricultural material was discussed. Solution pH was identified as a major factor influencing the sorption process. The high removal efficiency reported in the studies that adopted CPS showed its vast potential in the elimination of contaminants from water sources. Also, the regenerative potential of the adsorbent over several cycles indicated its long-term use. The economic feasibility and environmental sustainability afforded by using CPS chart a path for further investigation into the use of other low-cost agricultural materials in the elimination of environmental contaminants. © 2023 The Authors. *Journal of Chemical Technology and Biotechnology* published by John Wiley & Sons Ltd on behalf of Society of Chemical Industry (SCI).

Keywords: adsorption; Carica papaya seed; heavy metals; microbes; trace organic chemicals; water

INTRODUCTION

The universal importance of good-guality water and the continuous development of industrialization has placed tremendous pressure on water resources, which consequently results in water pollution emanating from organic, inorganic and microbial contaminants. Hence the need to treat contaminated water has become a global concern because of recent reports on the levels of these contaminants.¹ Water pollution can be defined as any alteration in the biological, chemical and physical features of water that leaves a destructive consequence on the health of humans.² Inorganic and organic contaminants, especially heavy metals, radionuclides, nutrients and trace organic chemicals (TrOCs), have been discharged into water bodies from different anthropogenic sources ranging from agricultural activities, leakage of leachate from landfills, and municipal and industrial effluent discharge.³⁻⁹ TrOCs include herbicides, pharmaceuticals, antibiotics, personal care products, steroid hormones, parabens, anions, and toxins produced by bacteria. Some of these contaminants are toxic to aquatic and human life at low concentrations owing to their mutagenicity, carcinogenicity and teratogenicity.¹⁰⁻¹³ The associated effects of these contaminants include impaired reproductive ability, disrupted endocrine system and sometimes death. $^{\rm 14-17}$

Heavy metals, which include lead, mercury, copper, zinc, chromium, cadmium and arsenic, are non-biodegradable, which leads

- * Correspondence to: AA Bayode or MO Omorogie, Department of Chemical Sciences, Faculty of Natural Sciences, Redeemer's University, PMB 230, Ede 232101, Nigeria. E-mail: bayodea@run.edu.ng (Bayode); omorogie@run.edu.ng, mo.omorogie@tum.de (Omorogie)
- a Department of Chemical Sciences, Faculty of Natural Sciences, Redeemer's University, Ede, Nigeria
- b Hydrochemistry Research Laboratory, Department of Chemical Sciences, Adekunle Ajasin University, Akungba Akoko, Nigeria
- c Department of Chemistry, Obafemi Awolowo University, Ile-Ife, Nigeria
- d Environmental Science and Technology Research Unit, African Centre of Excellence for Water and Environmental Research (ACEWATER), Redeemer's University, Ede, Nigeria
- e Chair of Urban Water Systems Engineering, School of Engineering and Design, Technical University of Munich, Garching, Germany

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to their accumulation in the environment⁴ and may be passed along the food chain.¹⁸ The term refers to metals with a specific gravity greater than 5 g cm⁻³ and is widely associated with pollution and potential toxicity.^{19,20} Some are toxic to humans and aquatic life at higher concentrations, causing, for example, kidney damage, bronchial dermatitis, and liver and brain problems.²¹

Furthermore, organic pollutants comprise two categories: one is hardly or non-biodegradable (e.g., TrOCs), while the other includes oxygen-demanding substances, which are susceptible to degradation by bacteria. The latter generally emanate from distilleries, breweries, tanneries, paper and pulp industries, municipal sewage, domestic outlets and canning industries. They contain enormous quantities of biodegradable organic compounds in dissolved colloidal and suspended forms. In stark contrast, synthetic organic compounds comprise volatile organic compounds, synthetic pesticides, paints, plastics and pharmaceuticals, and they pose severe risks such as convulsions, nausea and chemical burns.^{2,22}

On the other hand, biological contaminants (bacteria, viruses and protozoa) usually enter water resources from human and animal faecal waste containing these microorganisms. According to a report by the World Health Organization, at least 2 billion people use drinking water sources contaminated with faeces, which is the leading cause of diseases such as diarrhoea, cholera, dysentery, typhoid and polio.²³ The pathogens must be killed, deactivated or removed from the water to make it safe for drinking. The United States Environmental Protection Agency regulated that the total coliforms in drinking must be less than 5% in a month for the water to be safe for consumption.^{4,24}

The need to remove these contaminants from water is urgent if the water is to be used for drinking. Over the years, several treatment techniques have been adopted by several researchers, which include filtration,^{25,26} nanofiltration,²⁷ reverse osmosis,²⁸⁻³¹ electrophoresis, chelation and lime softener,²⁷ coagulation, flocculation,²⁸⁻³¹ ozonation³²⁻³⁴ and advanced oxidation processes (AOPs).³⁵⁻⁴⁵ These conventional methods have been observed to have some hitches, such as the release of by-products, ineffectiveness against some contaminants and cost-effectiveness in management.⁴⁶ Among the physicochemical processes that have proved effective for water treatment, adsorption has been adopted to tackle most of the challenges, which is easy in operation and inexpensive. The process of adsorption is capable of eliminating both insoluble and soluble organic pollutants.47-49

Biogenic adsorbents have been synthesized from biodegradable agricultural waste such as *Carica papaya* seed (CPS),⁴⁸ plantain peel,^{52,53} orange peel,⁵⁴⁻⁵⁶ carrot powder,⁵⁷ coconut shell powder,^{58,59} rice husk,^{60,61} sugar cane bagasse,^{62,63} corn silk^{64,65} and inorganic materials like clay minerals to improve efficiency or adsorption capacity. These materials act as a support in adsorption and, most significantly, in synthesizing photocatalysts to offer a higher specific surface area and strong adsorption affinity towards the pollutants.⁶⁶

CPS, an agricultural waste product from pawpaw, has been explored as an adsorbent (activated carbon)⁵⁰ or component of composites used either as adsorbents or photocatalysts for the removal of contaminants in water. CPS has been extensively used as an efficient adsorbent for the purification of water polluted by dyes,^{50,67-70} pharmaceuticals,⁷¹⁻⁷³ heavy metals⁷⁴⁻⁷⁷ and anions.⁴ Despite the abundance of research studies on the use of CPS in the remediation of environmental contaminants, there is a lack of recent comprehensive reviews on the subject. This present review is focused on filling these knowledge gaps. This study discusses the characterization of Carica papaya through different analytical techniques. In addition, the removal of inorganic, organic and microbial contaminants with CPS and all the modified materials that stem from it over the years are elucidated.

CHARACTERIZATION OF CPS

Characterization of materials (photocatalyst or adsorbent) for the sequestration of contaminants in water is very significant. It helps to confirm the functional moieties and all other properties of the adsorbents and photocatalysts, which in turn aids the removal of various pollutants. Some characterization analyses carried out on the biomass and biomass-based materials as adsorbents or photocatalysts are X-ray fluorescence (XRF), transmission electron microscopy (TEM), X-ray diffraction (XRD), Fourier transform infrared (FTIR), point of zero charges (pHpzc), Brunauer-Emmett-Teller (BET) surface area, scanning electron microscopy (SEM), and proximate and elemental analysis. Based on available evidence in the literature, the characterization of CPS is shown in Fig. 1.

The BET surface area of CPS is small (11 m² g⁻¹), which is one of the main characteristics of agrogenic or biomass materials.⁷⁸

FTIR is used in identifying absorption bands corresponding to the functional groups present in a material.⁷⁹ In a study, CPS spectra showed the presence of functional moieties like -OH associated with hydroxyl in the carboxylic and phenolic present in the seed.⁶⁶ The N–H stretching vibration associated with an *N*-substituted amide occurred around 3480 and 3440 cm^{-1} , while the C-H stretch vibration of methyl and methylene groups occurred around 2930 cm⁻¹. The presence of a large number of functional groups in the spectra of CPS is an indication of its potential to remediate a wide variety of contaminants/ pollutants.⁸⁰

The SEM image of the outer rind and CPS powder, as reported by Unuabonah et al,⁶⁶ showed that it has an irregular, rough surface structure and is porous, which could be a significant characteristic in the removal of contaminants from water. The pHpzc of CPS was reported to be 7.0.

CPS contains over 50% and 30% of carbon and oxygen, respectively, as shown in the elemental analysis in Table 1: CPS also exhibits a certain amount of H, S and N, indicating a significant fraction of organic materials. The XRF analysis is summarized in Table 2, showing the presence of some elements (Si, Al, K, Fe, Cu, Pb, Mo, Ni and Cd).

Upon modification of CPS with other adsorbents or metals or minerals, the functionalities, surface chemistry and properties change. The interaction between CPS and the material used to modify it can be tracked using various characterization techniques.

Bayode and her colleagues modified CPS with kaolinite clay and ZnCl₂ and NaOH to yield PS-HYCA. The interaction of the different components was confirmed using characterization tools including FTIR, SEM, energy-dispersive X-ray spectroscopy (EDX), TEM, selected-area electron diffraction (SAED) and electron mapping.⁸²

The FTIR spectra in Fig. 2(A) show the interaction between the papaya seed, kaolinite clay and zinc chloride. For the Carica papaya spectra, different peaks at 3409, 2922, 2448 and 1707 cm⁻¹ suggest the presence of -O-H in the seed, -C-H stretch vibration of methyl and methylene groups, and -C=O stretch vibration of carbonyl double bond resulting from the carboxyl group and amide band I of protein, respectively.⁶⁶ The spectra of PS-HYCA showed a significant interaction between the





Figure 1. (A) FTIR spectra, (B) XRD patterns of kaolinite clay, pure Carica papaya seeds and HYCA, (C) pHpzc and (D) SEM image of Carica papaya seed.⁶⁶

kaolinite clay and CPS, and there was a shift in the peak of -C-O stretching from carboxylic acids and their salts from 3406 to 3416 cm⁻¹. The formation of new peaks at 2360 and 1600 cm⁻¹ is indicative of -C=C=C-, -C=C=O-, -C=C- stretching vibrations and -OH of water.⁸¹ The presence of -Si-O-Zn stretching vibration was observed at 1086 cm⁻¹ and the presence of ZnO was observed at 462 cm^{-1.82} All these suggested the interaction of all the components to give PS-HYCA.

Figure 2(B) shows a high-resolution TEM image of PS-HYCA, confirming the agglomeration of zinc oxide on the surface of the composite, which is also confirmed by FTIR. The field-emission SEM image showed that the particles are irregular in shape and small in size (Fig. 2(C)). The electron mapping image and EDX confirmed the presence of all the elements (Ca, Mg, Zn, Si, O, Na, Cl, Fe) present in the composite (Fig. 2(D,E)). SAED confirmed that the composite was crystalline; this was a result of the kaolinite clay present in the composite (Fig. 2(E)).⁸²

Characterization of the CPS and modified CPS is not only crucial because of the useful information it provides for sorption studies, but it is also important because the adsorbent is susceptible to decay. Hence the need arises to determine the state and/or quality of the adsorbent before its use for the abatement of pollutants using suitable characterization techniques.

Table 1.	Elemental analysis data of Carica papaya seed ⁶⁶	
Element	(%)	
с	54.9 ± 3.6	,
Н	6.3 ± 1.2	
N	4.6 ± 1.1	
S	1.9 ± 0.3	
0	32.3	

CPS FOR THE REMOVAL OF INORGANIC CONTAMINANTS

The removal of inorganic substances continues to generate significant attention. Much research focus is directed at improving the adsorption capacity of CPS. For example, feldspar, an anhydrous three-dimensional aluminosilicate, was blended with CPS via simple calcination for the removal of Pb²⁺, Cu²⁺ and Cd²⁺ from water. Calcination caused a reduction in the specific surface area of the resulting composite (8.9 m² g⁻¹) compared with pristine feldspar (15 m² g⁻¹). However, the calcination of CPS with feldspar caused an increase in the cation-exchange capacity of the composite (40 meq 100 g⁻¹). Thus, there was an improvement in adsorption capacity of 17.1, 9.91 and 11.3 mg g⁻¹ for Pb²⁺, Cu²⁺ and Cd²⁺ respectively.⁸³ Similarly, CPS was modified using bentonite clay, a mineral used for the removal of Pb²⁺, Cu²⁺ and Cd²⁺ from

Table 2.	X-ray fluorescence (XRF) of <i>Carica papaya</i> seed ⁶⁶	
Oxide		(%)
SiO ₂		0.13
AI_2O_3		0.05
Na ₂ O		0.05
CaO		1.11
Fe_2O_3		0.05
CuO		0.01
K ₂ O		4.18
SO3		1.49
P_2O_5		1.74
PbO		0.01
MoO ₃		0.01
NiO		0.004
CdO		0.013



Figure 2. (A) FTIR, (B) HR-TEM, (C) FE-SEM, (D) electron mapping, (E) EDX and (F) SAED of kaolinite-modified Carica papaya seed (PS-HYCA).⁴⁷

water.⁸⁴ The modification did not result in an enhanced removal capacity but improved the composite mechanical stability. In another study, the mineral kaolinite clay/Carica papaya composite was prepared by simple calcination for the removal of Cd^{2+} , Ni^{2+} and Pb^{2+} from water.⁶⁶ The modification caused a reduction in the composite average particle size from 200 to 100 µm and an increased affinity for Cd^{2+} , Ni^{2+} and Pb^{2+} due to the enhanced cation-exchange capacity. This result proved that kaolinite clay modification was better than bentonite clay modification as there was an increase in the removal of the heavy metals Cd^{2+} , Ni^{2+} and Pb^{2+} .

The defatting of CPS was also explored for the removal of Pb²⁺ and Cd²⁺. It caused the appearance of -C=0 and -OH functional groups on the surface of the adsorbent and enhanced reactive sites (143 m² g⁻¹).⁷⁶ The adsorbents were employed to remove Pb²⁺ and Cd²⁺ from water in a single and a binary system. In the single system, the adsorption capacity was 1666 and 1000 mg g⁻¹ for Pb²⁺ and Cd²⁺, respectively. However, the adsorption capacity was <8.93 mg g⁻¹ in the binary system of Pb²⁺/Cd²⁺. This implies that competitive adsorption for the active site was unfavourable for the adsorbent. In another study, defatted CPS was used to remove the heavy metal ions Cu²⁺ and Pb²⁺. The experimental adsorption capacity obtained was reported as 17.3 and 56.0 mg g⁻¹ for Cu²⁺ and Pb²⁺ respectively. The equilibrium adsorption data fitted the Langmuir isotherm model. The removal efficiency of the adsorbent was also reported to be 97.6%.⁸⁵

In another recent study, CPS was employed as an adsorbent for the removal of Zn^{2+} . The adsorption capacity was reported to be 19.9 mg g⁻¹ and the equilibrium adsorption data fitted the Langmuir isotherm model. In contrast, the kinetic data conformed to the pseudo-second-order kinetic model, suggesting the chemisorption of Zn^{2+} onto CPS.⁷⁴ Similarly, Cr⁶⁺ was removed by raw papaya seed powder (RPS). The maximum adsorption capacity was reported to be 106 mg g⁻¹ and the optimum removal was achieved in the acidic pH region of 1.0. The kinetic and experimental equilibrium data fitted the Langmuir and pseudo-second-order models, respectively.⁸⁶

Modification of CPS as an adsorbent for the removal of inorganic contaminants from water can also be achieved via chemical means. Carboxyl and amino functional groups were grafted onto CPS,⁸⁷ and the adsorbent was used to remove Hg^{2+} . Ethylenediaminetetraacetate (EDTA)-functionalized CPS is shown in Fig. 3. The value of the adsorption capacity was 18.2 mg g⁻¹, and the removal of Hg^{2+} was caused by the ion exchange of Na⁺ by Hg^{2+} and interaction resulting from the presence of the carboxylic group on the surface. The removal capacity for Hg^{2+} depends on the initial solution pH.

A blend of CPS, *Musa paradisiaca* and ZnCl₂ was functionalized with an organosilane to remove phosphate from an aqueous solution.⁸⁸ The chemical functionalisation with organosilane caused the introduction of $-C=NH^+$ on the surface of the composite. The presence of the $-C=NH^+$ functional group brought about a 40-fold increase in the adsorption capacity of the composite for the removal of phosphate in an aqueous solution when compared with the hybrid (CPS, *Musa paradisiaca* and ZnCl₂) without amino functionalization. Thus, functionalizing CPS can increase the adsorption capacity of the resulting adsorbent.

Papaya seed charcoal (PSC1) was used for the removal of Pb²⁺ ion. PSC2 was prepared by mixing CPS with concentrated H_2SO_4 for activation and carbonization. The black slurry formed after the procedure was further heated in a hot-air oven at 150 °C for 24 h, washed and dried. The experiment was carried out in a batch system using PSC1 and PSC2. The adsorption capacity was reported to be 181 and 238 mg g⁻¹ for PSC1 and PSC2,



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Figure 3. EDTA-functionalized Carica papaya seed for the removal of Hg^{2+,87}

respectively. The pseudo-second-order model fitted all the experimental kinetic data. The study further evaluated the desorption efficiency of PSC1 and PSC2, and it was reported to be used for four adsorption–desorption cycles.

CPS has been adopted to support clay minerals to mitigate drawbacks such as heterogeneous functional surfaces, fragile molecular structure and low cation-exchange capacity. Composite materials have been employed for water treatment processes; CPS-modified feldspar clay for the adsorption of Pb²⁺ and Cu²⁺ in water improved the adsorption capacity of feldspar clay from 18.3 to 45.8 mg g⁻¹ for Pb²⁺ and from 12.6 to 46.2 mg g⁻¹ for Cu^{2+.89}

Despite the enhanced removal efficiency of inorganic contaminants exhibited using CPS in the adsorption process, calcination is an influential factor in adsorption efficiency. The calcination process could not only remove volatile substances and impurities from the adsorbent but also enhance the adsorbent's adsorption affinity.^{66,90} Nevertheless, a suitable calcination temperature for maximum adsorption capacity would be required for the optimum sequestration of these inorganic contaminants. Furthermore, the use of CPS in the removal of metal ions has been aided by several modifications of the adsorbent. While some of these modifications have significantly improved the adsorbent's cation-exchange capacity, mechanical stability and consequently its adsorption capacity, some of the studies indicated that the adsorbent is not the only influential factor aiding the adsorption process. Another factor implicated to influence the removal of the metal ions is the pH of the solution. Each metal ion has a characteristic pH at which its adsorption occurs. Generally, for cationic metals, an initial solution pH in the basic region (high pH values) facilitates adsorption, whereas an initial solution pH in the acidic region (low pH values) ensures greater adsorption.⁹¹

CPS FOR THE REMOVAL OF ORGANIC CONTAMINANTS

CPS-based materials in composition with natural or synthetic or metal oxides have found significant applications in the removal of organic and microbial contaminants, which showed improvement in the adsorption capacity. The use of CPS in the removal of organic contaminants is presented in Table 3.

CPS has been used as a natural biosorbent to remove tannery dye (Direct Black 38) from an aqueous solution, which was prepared by drying fresh seeds in the oven at 80 $^{\circ}$ C for 12 h, followed by grinding in a knife mill. Its characterization indicated that it

possesses macro/mesoporous texture, large pore size and a surface containing various organic functional groups, which are suitable properties for adsorption. The equilibrium data fit best in the Langmuir isotherm, showing a high adsorption capacity of 440 mg g^{-1} , with its kinetic data suitable for pseudo-first-order and pseudo-second-order, intraparticle diffusion and Boyd models. Another researcher explored the use of defatted CPS for the adsorption of methylene blue (MB); it was prepared by crushing fresh CPS and treated in a Soxhlet extractor using hexane to obtain defatted Carica papaya seeds (DCPS). The adsorption capacities were 1250 and 769 mg g^{-1} , respectively. The absence of some compounds such as unsaturated ketone, keto (enolic) esters and lactones, guinones and carboxylic acids in the DCPS was reported, while the presence of carboxylic, phenolic and lactone functional groups was confirmed by surface chemistry studies. The pore size and diffusion mechanisms played a vital role in the adsorption of MB in an aqueous solution.⁵⁰

Furthermore, methylene blue and basic fuchsin (BF), which are cationic dyes, are removed from the aqueous solution by DCPS, and prepared by treatment of ground dried papaya seeds with *n*-hexane. Compared to non-defatted CPS, the adsorption capacities of both dyes increased from 93.2% to 97.2% for MB and from 88.3% to 95.1% for BF within the pH range of 2.0–3.0, indicating stability when varying the pH from 3.0 to 11.0. The kinetics and adsorption isotherm followed the pseudo-second-order rate law, while the adsorption parameters were in good agreement with the Freundlich isotherm for MB and Temkin isotherm for BF.⁹² These results proved that defatted CPS was more efficient than non-defatted CPS.

In another investigation, CPS was used to remove MB in an aqueous solution, prepared by crushing oven-dried fresh CPS in a knife mill and sieving the resulting material. The CPS was observed to have a mesoporous to macroporous pore size and a specific surface area sufficient for the adsorption of MB. The equilibrium data best fit into the Langmuir isotherm, showing a maximum adsorption capacity of 638 mg g⁻¹. However, adsorption kinetic data were suitable for pseudo-first- and pseudo-second-order models. Hybrid clay (HYCA) was synthesized by mixing kaolinite and CPS, which attained an adsorption capacity of 35.5 mg g⁻¹ for the removal of MB using a fixed-bed reactor system. The reusability test after approximately 20 min of MB-ladened HYCA reached at least 90% each cycle for five regeneration cycles, which allowed the material to be reutilized.⁹³

Table 3. Carica papaya seed adsorbents for the removal of organic contaminants in water									
Composite	Contaminant	Adsorption capacity (mg g^{-1})	References						
Fe ₂ O ₃ -PSP	Congo red	217	70						
Formosa papaya seed powder (FPSP)	Crystal violet	86.0	69						
КРА	lvermectin	105	73						
Papaya seed	Methylene blue	637	101						
CPS	Chlorinated phenol	29.4	95						
Alkali-soluble polysaccharides of CPS	Indigo carmine, Congo red	319	98						
		169							
PS-HYCA	Methylene blue	57.3	47						
CPS	Direct black 38	440	68						
CPS	Methylene blue	769	50						
DPS		1250							
KPZ	Ciprofloxacin	295	102						
	Tetracycline	229							
A-KPZ	Ciprofloxacin	142	102						
	Tetracycline	88							

(an endocrine disruptor) – was removed using CPS-modified kaolinite clay (CPK) and pinecone seeds, indicating specific surface area reduction in the composites. There was no structural modification in the lattice d-spacing, showing impregnation of the calcined CPS and pinecone seeds on clay surfaces and pores. Meanwhile, their cation-exchange capacities were increased by fourfold, suitable for adsorption and modification, improving the adsorption by 250% in CPK and 52% in modified pinecone seed, suggesting a more efficient biomass for CPS .⁹⁴

Chlorinated phenol was removed from the aqueous solution using carbon derived from CPS with varying dry-weight impregnation ratios of zinc chloride (ZnCl₂) to papaya seeds using a twostage generated atmosphere method. The prepared material showed an improved adsorption capacity of 91.6%, in compliance with the improved morphological structure, porosity and functional groups to enhance efficiency. Adsorption studies indicated that the material fitted well in the Langmuir isotherm ($q_{max} = 39.7 \text{ mg g}^{-1}$) and pseudo-second-order model ($q_e = 29.4 \text{ mg g}^{-1}$), suggesting the suitability of using CPS as a precursor to synthesize activated carbon.95 In another research study, an activated carbon/chitosan/CPS (CHASPAC) composite was synthesized by coating chitosan with activated carbon and CPS and the material used for the removal of MB. The maximum adsorption capacity of CHASPAC was observed to be 302 mg g^{-1} at pH 8.0 and 30 °C. Kinetic studies showed that it fitted pseudo-second-order and Elovich models, which explained the adsorption process better than the pseudofirst-order kinetic model. At the same time, the equilibrium data best fitted the Langmuir isotherm.⁹⁶ The adsorption of cationic dyes, (MB) and brilliant green (BG) was also studied using sorbents prepared from pulverized CPS and Citrus grandis rind. They were prepared by air drying at 70 °C in an oven, ground, sieved and characterized. The surface area and porosity indicate that CPS sorbent possesses a larger surface area, pore size and pore volume than pomelo rind sorbent. Hence the CPS sorbent has a high affinity for MB and BG dyes compared to pomelo sorbent, and the adsorption of both dyes is best fitted to the Langmuir isotherm model.⁹

The removal of indigo carmine (IC) and Congo red (CG) dyes was investigated using alkali-soluble polysaccharides of CPS (ASP) from single and binary solutions. The adsorbent was prepared by crushing for 5 days, dried, sieved, treated with 0.1 mol L⁻¹

NaOH and shaken using a magnetic agitator. The suspension was filtered after standing for 12 h and oven dried at 60 °C for 6 h, and ASP was obtained. The maximum adsorption capacities of CR and IC were reported to be 319/636 and 169/103 mg g⁻¹, for the single/binary mixture, respectively, and fitted in the Langmuir–Freundlich isotherm from the experimental data of both. The adsorption mechanism for the removal of the dyes was reported to be the electrostatic interactions between functionalities present on the surface of the adsorbent and dye molecules and showed good recovery ability after five cycles.⁹⁸

Papaya seed activated carbon was also investigated for the removal of methyl parathion, an insecticide, from an aqueous solution, which was prepared by washing, then dried, ground, stored and carbonised using sulfuric acid in a ratio of 4:3 and kept in an oven at 160 °C for a period of 12 h. It was then dried in an oven at 100 °C for 4 h and washed to a neutral pH. The prepared adsorbent exhibited high adsorption capacity due to the functional groups and electrostatic attraction between the activated carbon and the insecticide. The equilibrium data for methyl parathion adsorption on papaya seed activated carbon best fitted the Freundlich isotherm model.99 Kaolinite-biochar composite sorbents were prepared from kaolinite clay and biochar from CPS (KPA) and pinecone (KPC). The composites showed noticeable property modifications, but the kaolinite clay primary lattice structure did not undergo any changes. KPC showed better efficiency in the removal of the contaminant at low concentrations with good adsorption capacity; KPA exhibited better reuse efficiency than the KPC with 83.5% and 67.5% over three cycles, respectively, compared to the reuse efficiency of KPC, reported as 73.8% and 58.8%.73

In the search for clean-technology material, zinc-modified hybrid clay composite adsorbent was made from kaolinite clay and biomass (CPS) employing microwave oven carbonization for the removal of MB. The material was synthesized by combining kaolinite clay, ZnCl₂ and crushed papaya seeds in a ratio of 1:3:1 for PS-HYCA adsorbent. Characterization indicates no alteration in the structure of clay; hence total modification was observed and showed an increased efficiency of adsorption of MB, best achieved at pH 7.0–11.0. The materials were used for the treatment of MB in aqueous and textile effluents at time intervals of 15 min and 900 min, respectively.¹⁰⁰ The researchers reported



Figure 4. Schematic representation of the mechanism of adsorption of MB dye onto PS-HYCA. 82

that the mechanism of adsorption of the MB dye into the adsorbent PS-HYCA was by electrostatic interaction between the MB molecules and carboxyl ion on the surface of PS-HYCA, weak physical forces such as van der Waals and hydrogen bonding between the nitrogen atom of the MB and hydroxyl group on the surface of the adsorbents, and chemical interaction between the dye and the adsorbents, as shown in the schematic representation in Fig. 4.

Munagapati *et al.*⁷⁰ synthesized Fe_2O_3 –PSP (magnetite loaded with papaya seed powder) to remove CR dye. The experimental kinetic data fitted the pseudo-second-order model with maximum adsorption efficiency reported as 217 mg g⁻¹ at pH 5.0.

Munagapati stated that the mechanism of CR removal by Fe_2O_3 -PSP occurs in three stages, with the primary mechanism being chemisorption, followed by physisorption. Electrostatic interaction and hydrogen bonding also play an efficient role in removing CR by Fe_2O_3 -PSP.⁷⁰

Steroid oestrogens – another class of contaminant in the endocrine disruptor group – were removed from water using a new visible-light p-n ZnO/graphene oxide (GO) supported on clay and CPS. It was prepared by combining pure kaolinite clay, ZnCl₂ and crushed CPS in a weight ratio of 1:4:1 to obtain K-ZnO/C, while K-ZnO/C/GO was obtained by reacting 1 g graphene oxide. Photocatalytic studies showed the removal of oestrogens – oestrone (E1), 17- β -oestradiol (E2), oestriol (E3) and the synthetic oestrogen 17- α ethinyl oestradiol (E2) – to be >89% and as high as 98% in a single run, and no significant difference was reported in the competitive system. In actual wastewater samples, the efficiency was 63–78% oestrogen removal.¹⁰⁰ They reported that the carbonized CPS played a vital role in the degradation of the steroid oestrogen by acting as an electron sink, thereby delaying the electron–hole recombination, as shown in Fig. 5.

In a recent study by Alfred *et al.*, a new nanocomposite, Cu@ZnWO₄-K, was synthesized and used for the degradation of acetaminophen (ACT), ampicillin (AMP), and sulfamethoxazole (SMX) from water. Characterization confirmed the presence of Scheelite (CuWO₄) and Ferberite (FeWO₄) crystal phases in the composite. Application results proved the efficiency of Cu@ZnWO₄-K composite with a degradation efficiency of 100%, 83% and 68% for AMP, ACT and SMX molecules, respectively. The authors reported that in the hybrid system the AMP molecule was the most degraded, with efficiency >98%. A further study was performed to ascertain the reusability of the composite, and it proved to be stable even after five cycles, showing it to be a sustainable and efficient material.⁷¹ In a similar study, a solar-active photocatalyst, TZPP₅, was prepared from kaolinite clay, sodium tungstate, titanium dioxide and biomass, and used to investigate



Figure 5. The role of carbon from *Carica papaya* seed in the degradation of steroid oestrogens in water.¹⁴

the photocatalytic degradation of ampicillin (AMP), sulfamethoxazole (SMX) antibiotics and the antimalarial drug artemether (ART). Photodegradation study showed degradation of >90% with TZPP₅ for all the contaminants. A further investigation was performed to ascertain the mineralization level, and it was reported to be 80% and 50% AMP and SMX, respectively, after 60 min. By-products in the form of inorganic radicals indicated a lower value compared to the WHO permissible limit for drinking water. At the same time, a reusability study showed a decrease in photodegradation efficiency to about 60% after two cycles. It also suitable for the treatment of real-life samples from rivers, stream abattoir water, etc.⁷²

The use of CPS-based composites for the removal of organic contaminants from water resources seems to be widely marked by increased porosity, morphological structure and functional groups. This is likely responsible for the high removal efficiency exhibited by CPS-based composites. However, it would be essential to identify the reaction conditions necessary for optimum removal efficiency.

CPS-BASED COMPOSITES FOR MICROBIAL CONTAMINANT REMOVAL

CPS-based materials have been reported for disinfection purposes – hence their involvement in therapeutic purposes for the treatment of some diseases. Their reported antioxidant properties and the presence of some organic functional groups also enhanced their utilization in the adsorption of microorganisms.

In an interesting study by Unuabonah et al.,⁸⁸ hybrid clay composites prepared from kaolinite clay and CPS were modified with chitosan, alum (a coagulant), NaOH and ZnCl₂ using solvothermal and surface modification methods. The hybrid clay surface modified with chitosan composite proved to be the most efficient for sequestration of Gram-negative enteric bacteria. This composite adsorbent has a maximum for Vibrio cholerae after 120 min, Escherichia coli after ~180 min and Salmonella typhi after 270 min. The adsorption capacities of Ch-nHYCA1:5 composite adsorbent for the removal of E. coli, V. cholera and S. typhi were reported to be 103, 154 and 83.7 mg.g $^{-1}$, respectively. The efficiencies for removal of these materials went beyond the alert level of 500 colony-forming units (CFU) mL⁻¹ in drinking water for these microorganisms.⁸⁸ Metal-doped hybrid clay composite was prepared with kaolinite clay, CPS and ZnCl₂, for the disinfection of Gramnegative enteric bacteria. From physicochemical studies, the adsorbent was shown to be a bacteriostatic rather than a bactericidal agent. It had great disinfection efficacy against Gramnegative enteric bacteria (S. typhi and V. cholerae), with a breakthrough time of 400 and 700 min, respectively.²³

CPS, on a few occasions, has been explored as a carbon support for the preparation of catalysts for the degradation of contaminants in aqueous solutions. Photocatalytically modified clay nanocomposite from low-cost sources using kaolinite clay and CPS, doped with Zn and Cu salts via the solvothermal method, was used to treat a multidrug-resistant (MDR) strain of *E. coli* bacteria in aqueous solution. The characterization of the materials suggested that Cu and Cu/Zn doping introduced new phases into the crystalline structure of kaolinite clay, which contributed to lowering the band gap of the material (see Fig. 4). The experiment was carried out in the fixed-bed mode to disinfect water contaminated with *E. coli*. A breakthrough time was achieved at 25, 30 and 35 h for Zn-, Cu- and Cu/Zn-doped nanocomposites, respectively, Further studies using multidrug and multi-metalresistant strains of *E. coli* were carried out using nanomaterials and the breakthrough time was reduced significantly.¹⁰³ The removal of a multi-drug-resistant strain of *E.coli* by Cu and Cu/Zn doping was by different mechanisms such as electrostatic interaction, metal leaching and photocatalysis via the production of reactive singlet oxygen species that were capable of lysing the bacteria cell wall, leading to cell death, as seen in Fig. 6.

In a similar study, a delaminated photocatalytic composite (DPC) consisting of kaolinite clay, urea, CPS, CuCl₂ and ZnCl₂ was synthesized and explored for the photo-mineralization of MDR E. coli and its sulfonamide resistance genes in contaminated water samples. It was reported that DPC modified with two metals (Cu and Zn) reduced the amount of MDR E. coli and its sulfonamide resistance genes in contaminated water. The log reduction was reported to be >6 at 36 h in a double disinfection process in the presence of visible light. Ninety-five percent photomineralization of MDR E. coli and its genes was attained through the discharge of superoxide radicals (in dark/light). No bacterial regrowth was observed in treated water stored in the light/dark for 7 days.¹⁰⁴ DPC was also explored for photo-disinfection of the fluoroquinolone-resistant genes parE and gyrB, and it was shown to significantly reduce the amount of these genes present. DPC proved to be a stable photocatalyst as it was tested over 7 months and it gave optimal results, showing it to have the potential for commercialization.

In summary, the foregoing analysis of the reviewed studies has shown the efficiency of CPS and its composites in the removal of microbial, organic and inorganic contaminants. A comparison of the adsorbent's efficiency with other adsorbents (Table 4) particularly highlighted a maximum adsorption capacity with the use of CPS. The maximum adsorption capacity exhibited by CPS may have been influenced by the operational parameters of the adsorption process. For example, solution pH is a significant influential factor in sorption processes. Cationic dves are favourably adsorbed at high pH values, while anionic dyes are preferentially adsorbed at low pH values.¹⁰⁵ Although methylene blue and rhodamine blue are both basic dyes, the sorption of methylene blue exhibited a better adsorption capacity relative to rhodamine blue. The former was carried out under a solution pH of 12.0, while the latter was carried out at a solution pH of 2. This explained the marked differences in their sorption capacity because basic dyes are known to undergo preferential adsorption at a pH greater than 7. This phenomenon is due to the emergence of the negative charge on the adsorbent's surface brought about by a reduction in the positive charge in the interface layer at the high pH of the solution. In contrast, at a low solution pH, the positive charge on the solution interface grows and is induced on the surface of the adsorbent, thus leading to an increase in the sorption of anionic (acidic) dyes.¹⁰⁶ It is unclear whether CPS would exhibit maximum adsorption capacity if the experiments were carried out under the same operational parameters of pH, initial concentration, time and adsorbent dosage. Nevertheless, it suffices to say that CPS is a low-cost alternative to other adsorbents that were adopted for the abatement of different pollutants.

DESORPTION AND REUSABILITY OF CPS-BASED MATERIALS

In the development of materials, the long-term reusability of the materials is a critical factor. Similarly, the reusability of the materials, either adsorbents or photocatalysts, over a long reuse cycle for a batch process is highly desired. To reuse spent adsorbent

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Figure 6. The role of carbon from papaya seed in the degradation of E.coli.¹⁰³

materials, it is essential first to desorb the adsorbate from the surface or active sites to regenerate the adsorbent and restore its activity. An easily regenerated adsorbent is highly sought after because it makes the treatment process economically feasible, and disposing of spent adsorbent without regeneration could pose a severe threat to the environment.^{88,127} Furthermore, the desorption of adsorbates is also an excellent way to recover resources, as some of the desorbed adsorbates could also be reused.^{88,128,129} The desorption of adsorbate adsorbed on the surface of an adsorbent is principally dependent on the surface chemistry of the adsorption process viz-à-viz the adsorption mechanism. Desorption is usually achieved by carefully selecting a suitable solvent/solution that can reverse the adsorption process. However, precautions must be in place to ensure that after the regeneration process the solvents do not block or destroy the active sites.¹³⁰⁻¹³⁵

For this course, several reports on adsorption studies using papaya seed-based adsorbents have also reported the desorption of contaminants from the surface of prepared materials. For instance, Pavan et al. demonstrated crystal violet (CV) desorption percentage from the surface of papaya seed powder (FPSP) as a function of the eluent (CH₃COOH) concentration $(0.05-2.00 \text{ mol } \text{L}^{-1})$. The optimum desorption was obtained using 1 mol L^{-1} CH₃COOH, with a CV recovery of 96.5%. The material only lost 6% of its capacity after five adsorption–desorption cycles.⁶⁹ To estimate the recovery of adsorbed CR and indigo carmine IC on ASP, an adsorption/desorption cycle experiment was performed. The adsorption experiment was carried out at pH 2.0. Different solvents, such as 0.1 mol L^{-1} EDTA solution, 0.1 mol L^{-1} NaCl solution, ethanol and acetone, were used to ascertain the best for the desorption process. Results obtained showed the desorption of the adsorbed IC dye to be 99.13%, 79.13%, 61.85% and 19.04%, while that of CR was 58.31%, 24.42%, 37.98% and 32.61% in the first cycle using EDTA, ethanol, NaCl and acetone, respectively. The result shows that EDTA had the highest desorption capacity, due to its solid chelating effect. The kinetic results of the Elovich model for CR were confirmed from the result because the desorption efficiency for IC was more than that of CR. The regeneration investigation showed a negligible decrease in CR removal efficiency during the five cycles performed, while a steady reduction in the IC removal rate from 94% to 68% was observed. The desorption and reusability studies revealed that ASP has a recycling ability.⁹⁸

Meanwhile, for the adsorption of MB using HYCA, it was reported that the adsorption efficiency remained unchanged after the fifth cycle.⁹⁰ In addition, results from the study suggested that changes in the concentration of the desorption solvent, HNO_{3} , from 0.001 to 0.1 mol L⁻¹ do not change the desorption rate and amount of desorbed MB. However, a change in temperature from 30 to 50 °C resulted in a decrease in the amount of MB dye desorbed (very low, 36%) and the desorption rate. This is because the adsorption of MB dye on HYCA is likely endothermic, as suggested by the adsorption data result. However, in another study on the family of modified papaya seed-based hybrid clay adsorbents, the reusability of the prepared adsorbents MB removal over three cycles using 70% ethanol as the desorption solvent was reported. The results revealed a strong interaction between the adsorbents and MB dye, with a decreasing desorption rate as the cycle progressed. This suggests that 70% ethanol is not ideally suited for the adsorbents' recovery and reuse.47

The result is consistent with the observations of Idohou *et al*,⁹⁶ as seen in their study of the desorption and reusability of activated carbon/chitosan/CPS composite prepared for the removal of MB using NaCl solution and ethanol as desorbing solvents. Low desorption was observed, which could be a result of the strong interaction between the active sites on the surface of the adsorbent and the MB molecules. Furthermore, with NaCl (94% and 61%) and ethanol (55% and 45%) desorption efficiencies for ASP and CH, respectively, physisorption was suggested to dominate the adsorption process of the MB dye by the adsorbent.⁹⁶

With regard to adsorption of CR using magnetic loaded papaya seed powder, Munagapati *et al*⁷⁰ demonstrated that a 0.1 mol L⁻¹ solution of NaOH showed the highest desorption efficiency of 96.3%. In contrast, the use of NaCl, CH₃COOH, HCl and H₂O resulted in low desorption efficiencies of 72.6%, 54.2%, 38.3% and 24.8%, respectively. The experiment was conducted using

in water									
			Adsorption						
Adsorbent	Contaminant	Operational variable	capacity	Model	References				
Papaya seed	Methylene blue	pH = 12.0	638	PSO	101				
Rice husk	Tetracycline	pH = 5.5, conc. = 0.5–32 mg L ⁻¹	3.89–13.85		107				
Potato peel waste (activated carbon)	Diclofenac	-	74.0	PSO	108,109				
Canola biomass	Metronidazole	pH = 7.0, Conc. = 0–100 mg.L ⁻¹	21.4	PSO	110				
Coconut husk	Chromium(VI)	pH = 2.0	222		111				
AC–Mg	Fluoride	pH = 8.0, time = 240 min, ads.	36.6	PSO	112				
AC–Si–Mg–La		dose = 0.2 g L^{-1}	54.5						
Orange peel	Cadmium(II) ions	Time = 120 min, conc. = 240 mg.L ⁻¹ , ads. dose = 0.04 g L ⁻¹ , temp. = 45 °C, pH = 5.5	12.2	PFO	113				
watermelon rind (WMR)	Acridine orange	_	69.4	PSO	114				
Activated banana peel carbon (ABPC)	Rhodamine blue	pH =2.0, time = 140 min, ads. dose = 0.2 g L ⁻¹ , conc. = 10- 120 mg L ⁻¹	46.9	PSO	115				
Corn silk/Zeolite-Y (CS/ZY)	Cadmium(II) ions	pH = 5.0, time = 5.5–11.5 min, ads. dose = 0.0009 g L ⁻¹ , conc. = 2.5– 12.5 mg L ⁻¹	315	PSO	116				
Luffa cylindrica sponge	Lead(II) ions	pH = 6.0	75.8	PSO	117,118				
Nitrilotriacetic acid-functionalized	Lead(II) ions	pH = 4.0-5.0, ads. dose = 0.1-1.0 g L ⁻¹ ,	54.4	PSO	119				
Adansonia digitata (NFAD)	Copper(II) ions	conc. = $25-200 \text{ mg L}^{-1}$	9.35						
Adenopus breviflorus seeds	2-Chlorophenol	—	97.7	PSO	120				
Chinese yam peel-polypyrrole (CYP- PPy)	Congo red	Conc. = 100 mg L^{-1} , time = 120 min, ads. dose = 10 g L^{-1}	86.7	PSO	121				
Yam peel carbon Fe ₂ O ₃ -modified	2,4-dichlorophenoxyacetic	pH =2.8, conc. = 50–300 mg L ⁻¹ ,	76.9	PSO	122				
yam peel carbon	acid	time = 300 min	78.7						
Sisal waste	Ibuprofen	pH = 4.0–6.0, time = 1440 min	140	PSO	123				
	Paracetamol		124	PSO					
Pine wood chips	Salicylic acid	pH = 2.0, time = 960 min	22.7	PSO	124				
	Ibuprofen		10.7	PSO					
K ₂ CO ₃ -modified cork waste	Ibuprofen	pH = 2.0–4.0, time = 360 min	417	PSO	125				
ZnCl ₂ -modified waste apricot	Naproxen	pH = 5.8, time = 60 min	106	PSO	126				
PS-HYCA	Methylene blue	pH = 12.0, time = 120 min	353	FKN	47				
PP-HYCA			207	2-step					
PS-PP-HYCA			206	2-step					
Ch-Noyce, 1:5	V. cholerae	Time = 240/180 min	83.6	—	88				
	E. coli		103						

Abbreviation: —, not reported; PS-HYCA, papaya seed-modified hybrid clay; PP-HYCA, plantain peel-modified hybrid clay; Ch-Noyce, chitosan and alum-modified hybrid clay; AC-Mg, magnesium-modified activated carbon from coconut husk; AC-Si-Mg-La, magnesium, silicon, lanthanide-modified activated carbon from coconut husk; ads., adsorbent; conc., concentration; PSO, pseudo-second-order kinetic model; PFO, pseudo-first-order kinetic model; 2-step kinetic, two-step kinetic rate model.

100 mg L⁻¹ of CR reacted with 0.05 g adsorbent, followed by batch desorption tests with 0.1 mol L⁻¹ solution of various solvents (NaCl, NaOH, HCl, CH₃COOH and H₂O) under the same conditions. Further experiments with NaOH as the desorbing solution showed that increasing the NaOH concentration (0.1–1.0 mol L⁻¹) decreased the desorption efficiency from 96.3% to 29.7% because of active site degradation. This report is similar to that of Unuabonah *et al.*⁶⁶ for the desorption of phosphate-laden amino-functionalized papaya seed-based HYCA. Furthermore, with 0.1 mol L⁻¹ NaOH, a drop in adsorption efficiency was observed. This may be attributed to Fe₃O₄–PSP, which decreased CR efficiency because of the drop in the available binding sites, leading to a significant decrease in regeneration efficiency for continuous cycles.

Although most reports are on applying papaya seed-based adsorbents for organic molecules, especially dyes, there are also reports on the reusability of papaya seed-based adsorbents for the adsorption of metals in water. Such a study on the reusability of papaya seed-originated charcoal (PSC) for the adsorption of Pb²⁺ has also been reported.¹³⁶ The recovery process for Pb²⁺-ladened PSC with 0.2 mol L⁻¹ HCl for four adsorption–desorption cycles was studied. The results show that PSC removal efficiency ranged from 98.5% to 91.5% for Pb²⁺ from the first to the fourth cycle.¹³⁶ In addition, it was observed that there was no difference in uptake pattern of Ni²⁺ from the water before and after the regeneration process for papaya seed-based HYCA materials.⁶⁶

The regeneration capacity of modified papaya seed (CMPS) for the adsorption of Hg(II) using HCl, H₂SO₄ and HNO₃ treatment was evaluated by Yadav et al.⁸⁷ Hence 0.5 mol L⁻¹ HCl was effective as regenerating solvent compared to other types and concentrations of acid. After acid regeneration and neutralization with 0.1 mol L⁻¹ NaHCO₃, CMPS was further used in three successive cycles. After the fourth cycle, the adsorption capacity decreased to 6.8%. This study suggests that the modified papaya seed-based adsorbent holds great economic potential for several reuse cycles. Another study, by Unuabonah et al.,⁸⁸ demonstrated that phosphate-laden amino-modified papaya seed-based hybrid clay adsorbents were easily regenerated using 0.01 and 0.1 mol L⁻¹ NaOH in five adsorption-desorption cycles. Interestingly, all the adsorbents showed decreasing trends in their adsorption capacity over the different adsorption-desorption cycles; the adsorbents functionalized with amino-organosilane (NPP-HYCA and NPS-HYCA) retained 84.1% and 94%, respectively, of their adsorption capacity after five cycles using 0.01 mol L^{-1} NaOH, 37.5% and 35.5% for iPSHYCA and iPP-HYCA adsorbents.88

Desorption of ivermectin by shaking spent papaya seed-based kaolinite-biochar adsorbent with 10 mL ethyl acetate twice at 200 rpm for 2 h each and drying the adsorbent at 60 °C by three adsorption-desorption-reuse cycles was reported by Olu-Owolabi *et al.*⁷³ Results indicated a decline in the adsorption strengths of KPA and KPC to 83.5% and 73.8%, respectively, after the first cycle, and 67.5% and 58.8% after the second cycle.

The practical use of any adsorbent is largely dependent on its potential for use multiple times.¹³⁷ A general overview of the desorption experiments indicated that there were variations in desorption efficiencies and these variations were related to the choice of solvents employed. In some instances, the desorption efficiencies were as high as >90%, while, equally, it was as low as about 45% in other cases. Similarly, some adsorbents could be reused for about four or five cycles, while others could only be used for no more than two cycles. It would appear that there are several factors influencing the regenerative efficiency of the adsorbent, including the type of pollutant, eluent used and adsorbent type (modified or unmodified).

CONCLUSIONS

Although several forms of water pollution exist, pollution of water by microbes, organic compounds and heavy metals continues to generate global concern. Developing countries seem to predominantly be on the wrong side of this due to the lack of effective and efficient technologies to decrease these contaminants. Nevertheless, the use of low-cost agricultural materials such as CPS to remove these contaminants from water resources has been investigated by several researchers. These research studies have shown that CPS can effectively remediate these contaminants. Chemical modifications of CPS generally increased the maximum adsorption capacity of the adsorbent. The present review highlighted that the adsorption efficiency of CPS depends on factors such as pore size, pH, morphological structure and calcination temperature. Furthermore, composites based on CPS also exhibited increased adsorption efficiency in the reviewed studies.

Using low-cost agricultural materials is both environmentally sustainable and economically feasible for providing clean water. The viability of the use of these materials in natural water resources should be further investigated. In addition, future research should be directed towards improving the surface morphology of these agricultural materials before use for the abatement of contaminants. It is also recommended that other techniques, such as advanced oxidation processes, should be incorporated before adsorption to ensure the complete removal of these contaminants from water sources. Finally, the use of CPS paves the way for further research into the remediation potential of other agricultural wastes that are disposed of into the environment.

ACKNOWLEDGEMENTS

Dr Martins O Omorogie and Prof. Dr Brigitte Helmreich are immensely grateful to the Alexander von Humboldt Foundation and the Carl Friedrich von Siemens Foundation for the award of Research Fellowship for Experienced Researcher to Dr Martins O Omorogie (NGA-1201082-HFST-E). The Chair of Urban Water Systems Engineering, Technical University of Munich, Garching, Germany is acknowledged for all technical support. Dr Martins O Omorogie appreciates the Department of Chemical Sciences, Faculty of Natural Sciences, Redeemer's University, Ede, Nigeria, for Research Fellowship leave.

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