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Removal of Orthophosphates in Simulated Municipal Wastewater by Adsorption using Activated Carbon from Banana (*Musa acuminata balbisiana*) Pith Impregnated with Ca^{2+} from Eggshells

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Phosphate pollution in municipal wastewater is a critical environmental issue due to its contribution to eutrophication and associated public health risks. This study investigates the efficacy of activated carbon derived from Saba banana pith, both impregnated with calcium ions (Ca^{2+}) from eggshells (ABPC) and without impregnation (ABP), as adsorbents for the removal of orthophosphate. Characterization of the surface morphology revealed that ABPC exhibited a greater porosity compared to ABP, providing a higher density of active sites for phosphate adsorption. The optimal pH for orthophosphate removal was pH 6 for ABP, achieving a removal efficiency of 38.23 %, and pH 9 for ABPC, with a removal efficiency of 91.11 %. Using an adsorbent dosage of 0.5 g, ABP achieved a maximum phosphate removal efficiency of 37.64 %, while ABPC reached 91.18 %. Adsorption isotherm analysis indicated that the Langmuir model best described the adsorption behavior for both ABP and ABPC, suggesting monolayer adsorption onto a surface with uniform binding sites. Kinetic analysis indicated that the adsorption process aligns with the pseudo-second-order model, suggesting that chemisorption governs the rate-limiting step. The findings demonstrate that ABPC is a highly effective adsorbent for orthophosphate removal, achieving performance levels that meet the guidelines established by DAO 2021-19 for environmental discharge. This study underscores the potential of utilizing biowaste-derived activated carbon for phosphate remediation in wastewater treatment.

1. Introduction

Rapid urbanization, industrialization, and an increase in population growth leads to escalation of wastewater generation (Dutta et al., 2021). Municipal wastewater contains dissolved and suspended organic and inorganic solids, heavy metals and pathogenic and pathogenic microorganisms (Nguegang and Ambushe, 2024). Worldwide, about 3.8×10^{11} m^3 of municipal wastewater are produced each year, with expected increases of 24 % by 2030 and 51 % by 2050 (Tariq and Mushtaq, 2023). Elevated phosphate concentrations in municipal wastewater are a major contributor to eutrophication, a growing environmental issue that accelerates algal blooms, degrades water quality, reduces dissolved oxygen levels, and disrupts aquatic ecosystems (Erasga et al., 2024).

Among the various chemical, physical, and biological approaches for phosphate removal from water, adsorption has emerged as a highly effective method, particularly for eliminating phosphates present at low concentrations (Almanassra et al., 2021). Adsorption has shown greater potential for phosphate removal due to its cost-effectiveness, high efficiency, and operational simplicity (Zhang et al., 2022). The phosphate-enriched spent adsorbent can be repurposed as a fertilizer and soil amendment (Erasga et al., 2024), enhancing its value and promoting environmental sustainability through the recovery and preservation of this essential non-renewable nutrient. However, conventional adsorbents exhibit limited phosphate adsorption capacity due to surface

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negative charge and rapid site saturation, coupled with weak phosphate binding and poor selectivity, resulting in suboptimal performance that restricts their applicability in large-scale engineering operations (Du et al., 2022). In recent years, numerous studies have explored the use of agricultural wastes as precursors for activated carbon production, owing to their renewable nature, cost effectiveness, widespread availability, and reduced environmental impact (Neme et al., 2022). Banana pith (BP), the starchy inner core of the banana pseudostem (*Musa acuminata*), is an agricultural waste generated year-round yet commonly discarded without beneficial utilization (Hasan et al., 2020). Although it currently lacks economic applications, BP contains 32.3 % carbon (Kakoi et al., 2016), and can serve as a valuable precursor for activated carbon production. Activated carbon derived from agricultural waste tends to exhibit reduced adsorption efficiency due to its sensitivity to temperature and pH (Gopalan et al., 2022). Hence, chemical modification, particularly through impregnation with metallic cations such as Al^{3+}, Fe^{3+}, Ca^{2+}, and Mg^{2+}, can enhance the surface characteristics of porous materials by increasing its surface area and improving adsorption capacity for anionic pollutants such as orthophosphate (Nobaharan et al., 2021). Numerous studies have demonstrated the effectiveness of calcium-impregnated biochar, produced using economical calcium sources such as chicken eggshells or calcium salts in significantly enhancing phosphate adsorption capacities, with modified rape straw using eggshell- and $CaCO_3$-derived calcium (Cao et al., 2020), and ramie biomass using calcium chloride dihydrate (Liu et al., 2016), confirmed the potential of calcium-functionalized biochars as low-cost and efficient adsorbents for phosphate removal.
This study investigates the potential of activated carbon derived from saba banana pith, modified through calcium ion impregnation using chicken eggshells, as an effective adsorbent for orthophosphate removal from simulated municipal wastewater. Both unmodified and calcium-modified activated carbon were evaluated to assess the influence of pH, adsorbent dosage, adsorption isotherms, and kinetic behavior, providing insight into their performance and applicability in wastewater treatment.

2. Materials and Methods

2.1 Preparation of Saba Banana Pith

Saba banana pith was prepared following the method of De Sousa et al. (2019), with modifications. Pith was obtained by peeling the outer pseudostem of Saba banana trees (Biñan City, Laguna, Philippines), then washed with tap water, rinsed with distilled water, diced, and boiled in distilled water for 15 min. Samples were sun-dried for 3 d, then oven-dried at 110 °C for 45 min until constant weight, and stored in airtight containers for subsequent use.

2.2 Preparation of Calcium Solution from Chicken Egg Shells

The calcium solution was prepared from chicken eggshells following the method of Habeeb et al. (2017), with modifications. A total of 150 g of eggshells sourced from households and bakeries in Biñan, Sta. Rosa, and Muntinlupa (Philippines) were washed thoroughly, dried in an oven at 105 °C for 45 min, and then ground into a fine powder. The powdered eggshells were added to a 25 % (v/v) acetic acid solution at a ratio of 0.05 g/mL and stirred at 150 rpm for 5 h. The mixture was then filtered to remove insoluble membranes, and the calcium-rich filtrate was collected for use, while the solid residue was neutralized before disposal.

2.3 Preparation of Activated Carbons (ABP and ABPC)

The calcium impregnation and pyrolysis procedure were adapted from El-Maghraby et al. (2014) with modifications. A total of 500 g of prepared banana pith was immersed in 1 L of calcium solution for 72 h with intermittent stirring to facilitate uniform calcium diffusion and distribution. After impregnation, the material was filtered and oven-dried at 110 °C for 45 min until constant weight, then stored in an airtight container. Both the untreated and Ca^{2+}-impregnated banana pith samples were pyrolyzed at 600 °C for 1 h in a muffle furnace. The resulting unmodified activated carbon (ABP) and modified activated carbon (ABPC) were cooled in a desiccator to prevent moisture uptake.

2.4 Characterization of ABP and ABPC

The microporous structure and surface morphology of ABP and ABPC were examined using Scanning Electron Microscopy (SEM) at NASAT Labs, Muntinlupa City, both before and after adsorption. For analysis, 0.5 g of ABP (at pH 6) and 0.5 g of ABPC (at pH 9) were used post-adsorption. Pre-adsorption samples were placed directly into the specimen chamber, while post-adsorption samples were first dried on a hot plate.

2.5 Preparation and Characterization of Aqueous Phosphate Solution

A phosphate stock solution with a concentration of 280 ppm was prepared by accurately weighing 0.2800 g of reagent-grade potassium dihydrogen phosphate (KH_2PO_4) and dissolving it in a 1 L volumetric flask with distilled water. The stock solution was freshly prepared prior to adsorption experiments due to the 48 h holding time of orthophosphates without chemical preservatives. To simulate realistic municipal wastewater conditions, a 14 ppm phosphate solution was prepared by diluting the stock solution. This value was based on Parsons (2014), who reported 14.28 ppm of phosphate in untreated wastewater from a public market in Muntinlupa, Philippines. The actual phosphate concentration was verified using a HANNA Instruments Phosphate High-Range Checker® (HC-HI717), and the initial pH was measured with a calibrated pH meter.

2.6 Batch Adsorption Experiments

Using 100 mL of simulated municipal wastewater with a 14 ppm phosphate concentration, the effect of pH was examined by adjusting the solution to pH values of 6, 7, 8, 9, and 10. The tests were carried out with 0.5 g of either ABP or ABPC, agitated at 100 rpm for 60 min under ambient conditions. Adjustment to the desired pH was accomplished using 0.1 M NaOH or 0.1 M HCl. The final pH and residual phosphate concentrations were measured after the treated solutions were filtered. Using the optimal pH from this experiment, adsorbent dosage effects were studied by varying ABP and ABPC masses (0.1, 0.25, 0.5, 1.0, and 2.5 g) under the same conditions.

2.7 Adsorption Isotherms and Kinetics Experiment

The methods for orthophosphate isotherm and kinetic studies were adapted from Cao et al. (2020). The experiments utilized 200 mL of phosphate solutions with initial concentrations ranging from 2 to 10 ppm. Optimal pH and adsorbent dosages were applied, with stirring at 100 rpm and contact times ranging from 5-minute intervals (first 20 min) to 10-minute intervals until equilibrium. At each interval, 10 mL samples were filtered through 0.45 μm membranes and analyzed for phosphate concentration using a HANNA Phosphate High-Range Checker® (HC-HI717).

2.8 Statistical Analysis

A one-way Analysis of Variance (ANOVA) was conducted to evaluate the effects of pH and adsorbent dosage on orthophosphate removal efficiency at a 95% confidence level. Tukey's Honestly Significant Difference (HSD) test was subsequently applied to identify statistically significant pairwise differences among treatment means.

3. Results and Discussion

3.1 Characterization of ABP and ABPC

Scanning electron microscopy (SEM) was employed to qualitatively characterize the surface morphology of ABP and ABPC prior to and following the adsorption process. As shown in Figure 1a, the surface of ABP prior to adsorption appears rough and crystalline-like, with few visible pores, indicating a limited number of potential sorption sites. In contrast, Figure 1c illustrates that ABPC also displays a rough, crystalline texture but with a greater number of clearly defined pores, suggesting enhanced surface heterogeneity and a higher density of active sites. This increase in porosity supports the suitability of modified activated carbon for adsorption, as porous structures are known to facilitate effective contaminant capture by providing more accessible binding sites (Yang et al., 2017).

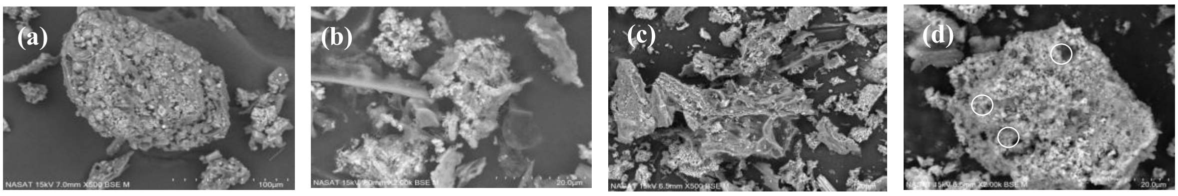

Figure 1: SEM micrograph of (a) APB before adsorption; (b)APB after adsorption; (c) ABPC before adsorption; (d) ABP-C after adsorption. (Condition: (a) x200, (b) x2000, (c) x500, and (d) x2000 magnification).

Post-adsorption micrographs reveal distinct morphological changes. In Figure 1b, ABP shows a wrinkled and smoother surface, with some pores obscured—likely due to phosphate occupying the available sorption sites. Similarly, Figure 1d shows that in ABPC, the adsorbed material is concentrated around the pores, partially filling or blocking them. Given that the simulated municipal wastewater contained only inorganic phosphate, the surface-bound contaminants are presumed to be phosphate species.

Comparative analysis of the two materials prior to adsorption indicates that ABPC has a more porous structure and a higher number of sorption sites than ABP. This structural advantage translates into a greater adsorption capacity. Furthermore, after adsorption, phosphate coverage on ABPC appears more uniformly distributed across the surface, while on ABP, adsorbates are localized mainly within or near specific pores. The reduced number of visible pores in ABPC after adsorption further confirms the extensive occupation of sorption sites, highlighting the material's improved performance due to calcium modification. Consistent with the research by Erasga et al. (2024), where banana peels served as the adsorbent, the SEM analysis showed similar surface morphological characteristics.

3.2 Effect of pH

The effect of pH on phosphate removal using 0.5 g of ABP and ABPC was evaluated across a pH range of 6 to 10, as presented in Figure 2. At 95 % confidence level, the findings suggest that ABP exhibited higher efficiency in orthophosphate removal under acidic conditions. At pH 6, the highest removal efficiency of 38.23 % was achieved, corresponding to a final phosphate concentration of 8.15 ppm and an adsorption capacity of 1.01 mg/g. In contrast, the lowest removal occurred at pH 10, with only 31.06 % removal and a final concentration of 9.10 ppm. This trend may be attributed to the presence of hydrogen ions (H^+) in acidic solutions, which can enhance phosphate adsorption by occupying active sites on the adsorbent. At pH 6, phosphate primarily exists as $H_2PO_4^-$, a monovalent anion capable of electrostatically interacting with positively charged sites on the ABP surface. The electrostatic attraction between $H_2PO_4^-$ and H^+ likely facilitates greater phosphate uptake. Under alkaline conditions, hydroxide ions (OH^-) compete with phosphate species, primarily in the form of HPO_4^{2-},for adsorption sites, leading to repulsion and reduced phosphate removal efficiency. The observed decrease in removal efficiency beyond pH 6 provides additional evidence of the dependence of the adsorption process on pH. Consistent with other studies, including those by Ouakouak and Youcef (2016), Nobaharan et al. (2021), and Erasga et al. (2024), it was found that phosphate adsorption on activated carbon is mediated by electrostatic interactions and ligand exchange, with optimal removal occurring at pH 2 – 6.

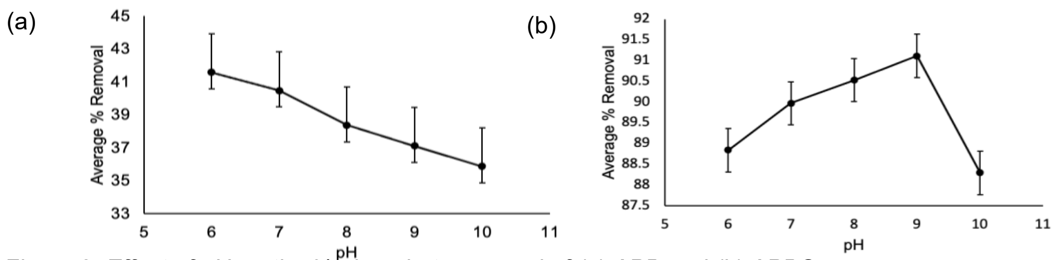

Figure 2: Effect of pH on the % phosphate removal of (a) APB and (b) APBC

Orthophosphate removal using ABPC was most effective at pH 9, achieving 91.11 % efficiency, with a final concentration of 1.18 ppm and an adsorption capacity of 2.40 mg/g. This can be attributed to the presence of Ca^{2+} on the adsorbent surface, which enhances phosphate adsorption. At pH 9, phosphate primarily exists as HPO_4^{2-}, which strongly interacts with Ca^{2+} due to their opposite charges and equal valency, promoting strong electrostatic attraction. In acidic conditions, H^+ ions may occupy active sites on ABPC, and $H_2PO_4^-$ binds to positively charged regions. Competition between Ca^{2+} and excess H^+ can limit adsorption. Since $H_2PO_4^-$ tends to associate more readily with H^+ than Ca^{2+}, phosphate removal at pH 6 is slightly reduced (88.84 %). At basic pH, although OH^- and HPO_4^{2-} both compete for adsorption under basic conditions, the strong ionic interaction between Ca^{2+} and HPO_4^{2-} still results in high phosphate removal at pH 9, wherein the orthophosphate concentration complied with the Department of Environment and Natural Resources (DENR) effluent standard of 4 ppm, and the final pH is within the allowable pH range of 6.0 to 9.5.

3.3 Effect of Adsorbent Dosage

The effect of increasing ABP dosage on orthophosphate removal was investigated, with results in Figure 3 showing increasing removal efficiencies. The highest removal of 37.64 % and an adsorption capacity of 1.00 mg/g were achieved at 0.5 g dosage, which was statistically significant. This trend can be attributed to the greater availability of adsorption sites, which is consistent with the findings of Ouakouak and Youcef (2016).

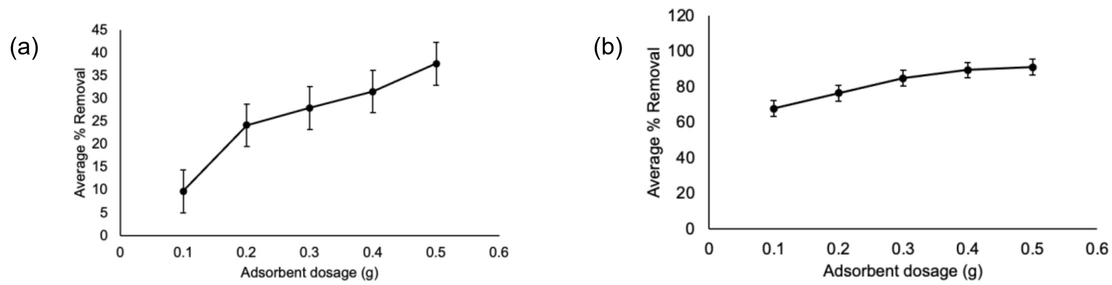

Figure 3: Effect of adsorbent dosage on the % phosphate removal of ABP (pH =6) and APBC (pH = 9)

However, none of the final concentrations using ABP met the DENR General Effluent Standards (GES) limit of 4 ppm. In contrast, ABPC achieved significantly higher removal, with 0.5 g yielding 91.18 % efficiency and an adsorption capacity of 2.48 mg/g. Dosage had a statistically significant effect on ABPC as well, and final concentrations at 0.2–0.5 g met the DENR GES, unlike the 0.1 g dosage. The improved performance of ABPC is due to both the increased number of adsorption sites and the presence of Ca^{2+}, which enables electrostatic and metal-ligand interactions with phosphate anions. Similar findings by Liu et al. (2016) and Erasga et al. (2024) support that surface modification of activated carbon with metal ions enhances phosphate removal through mechanisms involving both ion exchange and calcium–phosphate complexation.

3.4 Adsorption Isotherm

Adsorption isotherm models are widely used for describing the equilibrium behavior of adsorbate and adsorbent interactions. As shown in Table 1, phosphate adsorption onto both ABP and ABPC is best described by the Langmuir isotherm model, as indicated by the highest correlation coefficient, demonstrating an excellent fit. It assumes monolayer adsorption onto a homogeneous surface with uniform adsorption energies and no interaction or migration of adsorbed molecules across the surface (Wang and Gu, 2020). Similar observations were reported by Cao et al. (2020), who found that phosphate adsorption on calcium-modified biochars is predominantly governed by monolayer adsorption. The calculated R_L values for both ABP and ABPC fall within the range of $0 < R_L < 1$, confirming that the adsorption of orthophosphate is favorable. The phosphate adsorption capacity improved significantly with ABPC, which achieved a maximum of 25.00 mg/g compared to 14.93 mg/g for ABP. Similarly, Chen et al. (2022) reported a maximum capacity of 52.96 mg/g for Ca/Zn-loaded biochar. Although ABPC exhibited a lower capacity, it remains competitive due to its simpler synthesis, lower-cost precursors, and its ability to meet the DENR GES of 4 ppm at dosages of 0.2 – 0.5 g.

Table 1: Adsorption isotherm constants for ABP and ABPC

Isotherm Model	Langmuir				Freundlich		
Parameter	q_{max} (mg/g)	K_L	R_L	R^2	K_f (mg/g)	1/n	R^2
ABP	14.9254	0.0202	0.9593	0.9987	0.3063	1.0235	0.9884
ABPC	25.0000	0.2371	0.6676	0.9911	4.9729	1.0898	0.9730

3.5 Adsorption Kinetics

The adsorption kinetics of both ABP and ABPC are best described by the pseudo-second-order model, as evidenced by high correlation coefficients across all phosphate concentrations (Table 2). The excellent fit of the pseudo-second order model indicates that the adsorption process is predominantly controlled by chemisorption. Calcium impregnation significantly enhanced the surface characteristics and adsorption efficiency of ABPC, resulting in superior phosphate removal compared to ABP. These results affirm that chemical bonding—through ionic or covalent interactions—plays a critical role in phosphate uptake. The adsorption kinetics observed in this study are consistent with Erasga et al. (2024), Hasan et al. (2020) and Chen et al. (2022), who also reported chemisorption as the primary mechanism in similar adsorbent materials. This indicates a strong chemical interaction between phosphate ions and the functional groups on the adsorbent surface, supporting the proposed adsorption mechanism.

Table 2: Rate Constants of Pseudo-first Order and Pseudo-second Order Kinetic Model for ABP and ABPC

	ABP				ABPC			
C_o (mg/L)	Pseudo-first order model		Pseudo-second order model		Pseudo-first order model		Pseudo-second order model	
	k_1 (min^{-1})	R^2	k_2 (g/mg-min^{-1})	R^2	k_1 (min^{-1})	R^2	k_2 (g/mg-min^{-1})	R^2
2.1	0.3224	0.9635	0.3502	0.9849	0.4989	0.9176	0.1726	0.9843
4.1	0.2502	0.9759	0.1679	0.9851	0.2979	0.9710	0.0807	0.9888
6.1	0.2119	0.9764	0.1178	0.9883	0.3429	0.9437	0.0528	0.9895
8.1	0.2467	0.9713	0.0865	0.9834	0.2825	0.9330	0.0400	0.9675
9.9	0.2409	0.9683	0.0691	0.9880	0.2434	0.9034	0.0314	0.9938

4. Conclusions

The calcium impregnation process significantly enhanced the surface characteristics and adsorption performance of ABPC, resulting in substantially higher phosphate removal compared to ABP. At optimal conditions (0.5 g dosage and pH 9), ABPC achieved a final phosphate concentration of 1.20 ppm, meeting the General Effluent Standards (DAO 2021-19) for treated wastewater. These findings demonstrate that ABPC is a more effective adsorbent for orthophosphate removal from simulated municipal wastewater. The improved performance is attributed to the increased availability of sorption sites and the presence of Ca^{2+}, which facilitates strong chemical interactions with phosphate ions. Overall, ABPC shows great potential as a sustainable and efficient material for phosphorus recovery and wastewater treatment applications.

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