Highly Efficient Low-Concentration Phosphate Removal from Effluents by Recoverable La(OH)₃/Foamed Nickel Adsorbent

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ABSTRACT: Lanthanum-based materials have attained increasing attention because of their high adsorption property of phosphate ions and their environmental harmlessness. However, challenges still remain to improve the phosphate adsorption capacity and find suitable materials for the lanthanum attachment substrate. Nickel foam with characteristics such as excellent uniformity, large specific surface area, high porosity, and low conductivity is considered to be the alternative for the preparation of lanthanum-based adsorption materials. An efficient adsorbent foamed nickel-based La (OH)₃ nanowire was first prepared with a facile one-step electrodeposition method. The batch static adsorption tests of simulative wastewater (e.g., coexisting ions and solution pH values) were employed to investigate the phosphate adsorption kinetics and solution matrix effects of the materials. The results indicate that the composite exhibits fast adsorption kinetics within 30 min and high selectivity to phosphate under interference from competing ions. The pH value of wastewater has great influence on the absorption of phosphate, and optimal adsorption capacity can be achieved over a pH 4–6 range. Various findings revealed that the adsorption behavior of lanthanum hydroxide/foamed nickel [La(OH)₃/Ni] followed inner-sphere adsorption through the ligand-exchange mechanism. The prepared material is expected to be an enormous potential candidate for the removal of low-concentration phosphorus from effluents.

1. INTRODUCTION
Discharge of wastewater is on the increase with the development of industries. Phosphorus (P) is a main nutrient in the aquatic environment,¹ but excessive P released into the natural ecosystems through effluents can trigger severe eutrophication and make the ecosystem collapse.² People may suffer from hyperphosphatemia in the natural environment of excessive phosphorus; furthermore, hyperphosphatemia could lead to renal osteodystrophy, vitamin D metabolic disorders, and secondary hyperparathyroidism, which may influence phosphorus and calcium deposition.³ Therefore, particular requirements have been given for P emission to control water pollution.⁴ Previous reports revealed that the phosphorus concentration should not exceed 0.03 mg P L⁻¹ in rivers and streams.⁵ Hence, various treatment technologies for the removal of phosphate with high efficiency are urgently needed for alleviating aquatic ecosystem eutrophication, in particular, for the removal of relatively low-concentration phosphorus to satisfy strict wastewater treatment requirements.⁶⁷

Among several techniques, such as chemical precipitation, physical adsorption, and biological treatments, physical adsorption is a preferable approach to remove and recover phosphorus because of its excellent properties of environmental-friendliness, operational simplicity, and high efficiency at low phosphorus concentrations.⁸ In recent years, metal-based adsorption materials have been widely used for phosphate removal in wastewater, such as hydrous zirconia-modified zeolite,⁹ magnesium oxide-enriched biochar,¹⁰ and hydroxy-iron-aluminum pillared bentonites.¹¹ Most of these adsorption materials have exhibited high adsorption selectivity and capacity for phosphorus, and these research groups are mainly concerned when the initial concentration of phosphorus is over 20 mg L⁻¹, which does not represent the characteristic of real contaminated water. In general, the phosphorus concentration of wastewater treatment plants treating effluents is under a certain level (10 mg P L⁻¹).¹² Herein, it is of key importance to develop appropriate sorbents with highly efficient phosphate removal capacity at relatively low P levels.

Lanthanum (La) has a good binding capacity to phosphate and an especially low solubility product in a complex system containing lanthanum phosphate, which has been employed as phosphate adsorbents.¹³ Moreover, La could provide more coordination sites and shows excellent sensitivity to phosphorus even at micro levels.¹⁴ Hence, a combination of La onto the support materials enhances both adsorption kinetics and capture capacity.¹⁵ However, the adsorption materials containing higher lanthanum loading usually exhibited significant amounts of lanthanum leachate in solution during the phosphorus sorption
process, and the La usage efficiency was lower. To our knowledge, there are few reports for low-concentration phosphate adsorption with the La-modified materials. Therefore, in this study, it is imperative and of great significance to develop a porous and environmental-friendly support for lanthanum deposition that could promote the removal of relatively low-level P and improve the lanthanum utilization efficiency. The characteristics of nickel foam with excellent uniformity, large specific surface area, high porosity, and low conductivity underpin the ideal applicability of using it as a support for La. Based on the above hypothesis, herein, a simple one-step electrodeposition method is developed to synthesize new sorbent nickel-based lanthanum hydroxide nanocomposites and La(OH)₃ nanomaterials for further structural characterization and evaluation by scanning electron microscopy (SEM), X-ray diffraction, and infrared. Phosphate adsorption kinetics and solution matrix effects of the new materials were investigated by static adsorption tests of simulated wastewater (including coexisting ions and solution pH values). The adsorption kinetics of phosphate shows that the composite has high adsorption capacity for phosphate and rapid adsorption within 30 min and reaches equilibrium at 6 h, but too high pH will inhibit its ability to adsorb phosphate. The various findings revealed that the adsorption behavior of La(OH)₃/Ni followed inner-sphere adsorption through the ligand-exchange mechanism. The prepared material is expected to be a promising candidate to remove low-concentration phosphate from effluents.

2. RESULTS AND DISCUSSION

Lanthanum hydroxide was electrodeposited on the cathode through an important mechanism of base (OH⁻) electrostatic attraction in nitrate solution,¹⁶ and the possible cathodic reactions are

\[
\text{NO}_3^- + H_2O + 2e^- \rightarrow NO_2^- + 2OH^-
\]

\[
2H_2O + 2e^- \rightarrow H_2 + 2OH^-
\]

The above reaction results in a pH value increase near the surface of the foamed nickel electrode. The production of H₂ may favor the formation of La(OH)₃ nanowires. With the increase of the OH⁻ concentration, the electrodeposition process takes place on the surface of cathode-producing La(OH)₃ nanowires

\[
\text{La}^{3+} + 3\text{OH}^- \rightarrow \text{La(OH)}_3\downarrow
\]

In the present paper, the galvanostatic cathodic electrodeposition process of La(OH)₃ was conducted in a three-electrode cell using a constant current density of 4 mA cm⁻². Figure 1a exhibits the potential value as a function of the electrodeposition time. Considering a working voltage of −1.03 V, it reveals that the water reduction plays an important role in the production of OH⁻ at the employed current density. As shown in Figure 1a, after a sharp potential drop from the open-circuit voltage to −1.2 V (Ag/AgCl), the electrode voltage exhibits almost a linear increase during the electrodeposition process.

The phosphate removal from the sample effluent can be achieved by applying La(OH)₃/Ni through batch phosphate adsorption process as illustrated in Figure 1b. Such ease of adsorption for La(OH)₃/Ni enables the high removal efficiency of phosphate. Furthermore, the fabricated adsorption device can effectively separate lanthanum, which would reduce the potential secondary harm of lanthanum residues to certain aquatic animals.

Figure 1c shows the SEM image of La(OH)₃, which exhibits a uniform nanonetwork morphological characteristic. The higher magnification SEM image of the La(OH)₃ nanowires is displayed in Figure 1d, which reveals that the sample possesses...
a homogenous nanowire microstructure throughout the surface of foamed Ni, and the particle size of the adsorbent is around 40 nm.

The energy dispersive X-ray spectroscopy (EDS) spectrum in Figure 2a reveals the existence of elements La, O, and Ni in La(OH)$_3$/Ni. As shown in Figure 2b, the P element peak is due
to the efficient phosphate removal from effluents by the lanthanum hydroxide/Ni adsorbent.

The abundant surface functional groups of La(OH)$_3$/Ni were determined via Fourier transform infrared (FTIR) spectroscopy, which were recorded in a band from 400 to 4000 cm$^{-1}$. The results of FTIR spectra verified that phosphate was successfully removed by the adsorbent. As shown in Figure 2c, a new peak was located at 1062 cm$^{-1}$ corresponding to the asymmetric stretch $v_3$ vibration of HPO$_4^{2-}$ or H$_2$PO$_4^-$ for the lanthanum hydroxide/Ni upon phosphate adsorption. This observation agrees with the expected phosphate species at pH 7.0. Furthermore, the peak at 1109 cm$^{-1}$ was obviously weakened, which may be ascribed to the occurrence of ligand-exchange reactions, in agreement with the findings of previous studies, further demonstrating that OH$^-$ played major roles in the phosphate adsorption process.

The correlative surface micromorphology of La(OH)$_3$/Ni after P adsorption was confirmed by SEM and is displayed in Figure 2d, and it was noted that La(OH)$_3$ nanowires possess a relatively homogenous surface. The corresponding EDS elemental mappings of La(OH)$_3$/Ni determined the existence of Ni (blue zone), La (yellow zone), P (green zone), and O (red zone), which were uniformly distributed throughout the foamed nickel surface, in favor of phosphate adsorption.

The surface area was obtained according to the Brunauer–Emmett–Teller (BET) theory from the nitrogen adsorption isotherms for La(OH)$_3$/foamed Nickel. Figure 3 displays the pore size distribution curve of La(OH)$_3$/foamed nickel, and the corresponding isotherms were employed to study the pore structure. The isotherms (Figure 3a) suggested that large pores (>50 nm) and mesopores (2–50 nm) were present in the sample. The hysteresis loop recorded for the material represents type H3 according to IUPAC classification, suggesting that the sample possesses flat slits, cracks, and wedge structure. These properties were well reflected in the adsorption capacity. In conclusion, La(OH)$_3$/foamed nickel possessed an average pore size of 3.836 nm, a pore volume of 0.118 cm$^3$ g$^{-1}$, and a surface area of 27.297 m$^2$ g$^{-1}$.

The X-ray photoelectron spectroscopy (XPS) measurements were also conducted to further probe the possible phosphate adsorption mechanism onto La(OH)$_3$/Ni. Figure 4a displays the full-range XPS survey spectrum, suggesting the evidence of O, La, and P elements in La(OH)$_3$/Ni after phosphate adsorption. The appearance of a standard peak located at ~835.0 eV indicated the intercalation of La(OH)$_3$ on foamed nickel. Obviously, after the phosphate adsorption, the distinguished binding energy peak of P 2p in the sample is located at ~133.2 eV, demonstrating the formation of complexes between phosphate and La(OH)$_3$. The representative P2p spectrum obtained from purified KH$_2$PO$_4$ centered at the binding energy of 134.0 eV, an obvious reduction of 0.8 eV, which confirmed the formation of high affinities between La(OH)$_3$/Ni and phosphate.

As shown in Figure 4b,c, before the phosphorus sorption, the La 3d$_{5/2}$ characteristic satellite peaks are located at binding energies of 838.9 and 835.6 eV. The binding energies of the peaks commonly shift to lower values, which may be ascribed to a possible electron transfer from the valence band of the ligand atom to the 4f orbital of the La atom. Obviously, the apparent transformation of the proportion onto La 3d$_{5/2}$ double peaks can be clearly observed because of the phosphorus adsorption, and an obvious peak shifted to the lower energy level, indicating the stronger interaction and affinities between lanthanum and phosphate. Accordingly, the wide peaks can be assigned to double peaks, and the presented new peaks were located at 837.3 and 834.6 eV. The La 3d$_{5/2}$ spectra shift to lower values suggested stronger affinities by the metal hydroxide surface because of formations of fresh inner-sphere complexation. Distinct satellite peaks of La 3d$_{5/2}$ are centered at 852.2 and 855.7 eV (Figure 4d,e) after phosphorus adsorption, and the binding energies of La 3d$_{5/2}$ shifted to lower values, suggesting a possible electron transfer in the valence band of La 3d and the formation of La–O–P inner-sphere complexation. The results are in line with a previous publication of phosphorus removal with nano-La(III) (hydr)oxide-modified wheat straw.

Based on the binding energy of the various oxygen species, the O 1s spectra of La–Ni before and after phosphate adsorption could be divided into three overlapped peaks corresponding to the adsorbed water (H$_2$O), M–OH (hydroxyl bonded to metal), and M–O (oxygen bonded to metal), as shown in Figure 4f.g. As displayed in Figure 4h, the peak with a binding energy of 133.2 eV was clearly observed in La(OH)$_3$/Ni after phosphate adsorption, which is ascribed to the P 2p spectra, suggesting that the phosphate was successfully adsorbed on La(OH)$_3$/Ni.

In this paper, Lewis acid–base interaction, electrostatic interactions, and ligand exchange are the main mechanisms for removal of P. For example, the OH functional groups on the La(OH)$_3$/Ni surface were replaced with the P species. The HPO$_4^{2-}$ or H$_2$PO$_4^-$ functional groups anchor on the La(OH)$_3$/Ni surface. Herein, the potential bidentate inner-complexation...
Figure 4. XPS analysis of La(OH)₃/Ni. (a) XPS spectra before and after the P sorption survey scan. La 3d₅/₂ core-level spectra (b) before and (c) after P sorption. La 3d₃/₂ core-level spectra (d) before and (e) after P adsorption. O 1s core-level spectra (f) before and (g) after P adsorption. (h) P 2p core-level spectra.
and monodentate may form, which facilitate the phosphorus adsorption with La(OH)$_3$/Ni when at pH 7.0. The phosphorus adsorption mechanism may be proposed as shown in Figure 5, which is based on the results mentioned above.

![Figure 5. Hypothesized possible adsorption mechanism of phosphate onto La(OH)$_3$/Ni.](image)

Overall, the results of the FTIR study agree well with the XPS analysis, indicating the high removal efficiency of phosphate by the sample. The XPS and FTIR results also confirmed the ligand-exchange mechanism.

To control water pollution, there is a need for wastewater treatment plants to meet an acceptable discharge phosphorus concentration, which is usually set as 0.05 mg P L$^{-1}$. Therefore, to obtain information about whether the La(OH)$_3$/Ni adsorbent is efficient, we performed the whole static adsorption experiments in simulative wastewater containing two different P concentrations (5.0 and 10.0 mg P L$^{-1}$). By increasing the La(OH)$_3$/Ni dosage to 1.0 g L$^{-1}$, the phosphate concentration in the sample can be reduced from 2.0 to 0.04 mg P L$^{-1}$, which is a satisfactory low level of P in secondary effluents from the wastewater treatment plants in China. The results suggested that the obtained La(OH)$_3$/Ni provided enormous potential as an efficient and fast sorbent for low-concentration phosphate removal.

The phosphate concentration was obtained using the ammonium molybdate method (Figure 6a) and a UV/vis spectrometer (Lambda 25, PerkinElmer, USA). The phosphorus sorption kinetics of the La(OH)$_3$/Ni sorbent was studied at various initial concentrations; Figure 6b presents the corresponding results. La(OH)$_3$/Ni has a significant stronger adsorption capacity compared with Ni. It could be seen that phosphate adsorption amount of above 85% by La(OH)$_3$/Ni was acquired in the first 30 min and then adsorption equilibrium was reached at 1 h. The initial efficient adsorption capacity of La(OH)$_3$/Ni may be attributed to the lanthanum hydroxide of La(OH)$_3$/Ni, resulting in a very rapid formation of lanthanum phosphate. La(OH)$_3$/Ni exhibited higher equilibrium adsorption capacity and a fast initial adsorption ratio.

Moreover, in order to explore the rate-controlling step in the adsorption process, the phosphorus sorption pathway was investigated using the intraparticle diffusion model. Figure 6c indicates that the adsorption process was composed of two linearity correlation regions before sorption equilibrium, demonstrating that phosphorus removal with the La(OH)$_3$/Ni adsorbent was a two-step process. The first part was film diffusion, implying that phosphorus diffuses quickly across the solution to the external surface of the adsorbent particles, which may contribute to the momentary occupation of active sites by the adsorption of the surface. Then, step 2 manifests that the phosphorus anions diffuse relatively slowly into the micropores or mesopores of the interior surface, which also confirm that diffusion rate constant $k_{f1}$ is larger than $k_{f2}$. Consequently, intraparticle diffusion played dominant roles in the phosphorus sorption process by the La(OH)$_3$/Ni adsorbent.

As displayed by the fitting correlation coefficient ($R^2$) in Table 1, the adsorption kinetics exhibited that the phosphate adsorption of Ni fitted a pseudo-first-order model (Figure 6d), suggesting a physical adsorption process, while the adsorption processes of La(OH)$_3$/Ni fitted a pseudo-second-order model (Figure 6e), suggesting a chemical adsorption process.

In general, the pH value of solution may play important roles during the phosphorus adsorption process. The phosphate removal with La(OH)$_3$/Ni at various solution pH (4−12) and the corresponding effects of solution pH are exhibited in Figure 6f, which suggests that the P removal performance of La(OH)$_3$/Ni was highly dependent on the solution pH value. The efficiency of P adsorption varied significantly, and in alkaline condition, it dropped sharply with the increasing pH value. Similar trends are in line with previous reports. There is low P adsorption efficiency at high pH values, which may be ascribed to the increase of the competition of OH$^-$ for the adsorption active sites. Based on the above results, the optimum pH value was from 5.0 to 7.0. Herein, there is no need for the adjustment of pH values in actual or working sample solutions. All of the final pH values at the adsorption equilibrium were higher than the initial ones, indicating that the concentration of OH$^-$ was elevated. The OH$^-$, HPO$_4^{2-}$, and H$_2$PO$_4^-$ anchor on the surface of the sample.

Competitive adsorption tests are essential for an effective phosphorus sorbent as realistic wastewater is often a mixture of naturally occurring ions, including potentially competitive cations such as Ca$^{2+}$ and Mg$^{2+}$ and anions such as Cl$^-$, SO$_4^{2-}$, CO$_3^{2-}$, and NO$_3^-$. As presented in Figure 6g, Ca$^{2+}$, Mg$^{2+}$, Cl$^-$, SO$_4^{2-}$, and NO$_3^-$ exhibited negligible effect on the P adsorption capacity, indicating that such ions had low competitions to the phosphorus adsorption with the La(OH)$_3$/Ni adsorbent even at a high concentration (10 mM). Nevertheless, phosphate removal amount was reduced by 42.0% because of the existence of CO$_3^{2-}$. The decrease may be attributed to the $K_{sp}$ value of LaPO$_4$ (3.7 × 10$^{-23}$) which is higher than that of La$_2$(CO$_3$)$_3$ (3.98 × 10$^{-34}$), resulting in higher coulombic repulsive forces that facilitated the formation of La$_2$(CO$_3$)$_3$. The strong selectivity of La(OH)$_3$/Ni on the tolerance of coexisting species could favor the removal of phosphate, further providing great potential utilizations of the sample in treating the actual wastewater.

The phosphate removal experiment was conducted using La(OH)$_3$/Ni for six continuous cycles to evaluate its reusability. As shown in Figure 6h, the phosphate adsorption rate maintained above 96% during six recycles. The decrease of P desorption efficiency may be attributed to the strong bond between P and La(OH)$_3$. These results suggest that the sample possesses excellent continuous P adsorption performance, which may have great practical potential in consecutive treatment of wastewater.

3. CONCLUSIONS

In summary, we have successfully fabricated unique arrays of lanthanum hydroxide nanowires by galvanostatic cathodic electrodeposition process using nickel foam templates from nitrate bath. The prepared materials possess a homogeneous structure. The batch static adsorption tests of simulated secondary effluents were used to investigate the phosphate removal.
adsorption kinetics and solution matrix effects of the materials. The results indicate that the composite exhibits fast adsorption kinetics within 30 min and high removal efficiencies of phosphate under interference with various competing ions.
Simultaneously, the pH value of wastewater played an important role in the absorption process of phosphate, and a high and relatively stable phosphate adsorption capacity can be achieved at pH 4–6. Moreover, the P adsorption efficiency of the sample maintained approximately 96% after six consecutive recycles. High removal efficiencies of the La(OH)3/Ni adsorbent for phosphorus may be ascribed to inner-sphere adsorption through the ligand-exchange mechanism and outer-sphere adsorption through electrostatic interactions. The –OH functional groups on the La(OH)3/Ni surface was replaced with the P species. The HPO42− or H2PO4− functional groups anchor on the La(OH)3/Ni surface. This work suggested that the prepared La(OH)3/Ni materials present a great promise as a suitable adsorbent for efficiency low-concentration phosphate removal from effluents in practical applications.

### 4. EXPERIMENTAL SECTION

4.1. Materials. All reagents applied in this paper were of analytical grade and used as received. NH4NO3, H2SO4 (98%), La(NO3)3·6H2O, NaOH, C6H5OCH2OH, (NH4)2MoO4·O7, C2H8K2O5Sb2, KH2PO4 and HOCH2CH2OH were obtained from Sinopharm Chemical Reagent Co., Ltd. (Ourchem Shanghai). Foamed nickel was purchased from Shanghai Danqi New Material Science & Technology Department. Deionized (DI) water with a resistivity of 18.25 MΩ·cm−1 was applied to prepare all solutions in the whole experimentation.

4.2. Preparation of La(OH)3/Ni Foam. Herein, the novel La(OH)3 nanowire adsorbent was prepared on foamed nickel via an electrodeposition approach. The electrodeposition process of lanthanum hydroxide nanowires was conducted in aqueous 0.05 M La(NO3)3 solution with slight stirring by applying a constant current density of 4 mA cm−2 using an electrochemical workstation (CHI 660E, Shanghai Chenhua Instruments Co.). A traditional three-electrode system was used with the foamed nickel as the working electrode, a saturated Ag/AgCl as the reference electrode, and a Pt wire as the counter electrode.

4.3. Adsorption Experiments. The phosphate stock solution was prepared through dissolving KH2PO4 (AR, 99.5%) and through further dilution with DI water to obtain various ionic concentrations, which were employed to study the influence of competing ions on the sorption process. The sorption kinetics experiment was carried out in 1000 mL of phosphorus solution with concentrations of 5 and 10 mg L−1.

### 4.4. Characterization. The microstructural characterization and chemical compositions of the as-synthesized materials were analyzed using XPS (Thermo Scientific ESCALAB 250Xi instrument) and FTIR (BRUKER TENSOR 27, Germany). The morphological characterizations and elemental distribution of the as-prepared samples were studied with the aid of a cold field emission scanning electron microscope (Quanta FEG 250, United States) and EDS spectrum collected from the EDAx EDS element (AMETEK, United States). Nitrogen adsorption/desorption isotherms were employed to characterize the textural properties of the La(OH)3/foamed nickel adsorbents at −194.85 °C. The surface area, pore volume, and pore diameters of the sample were obtained using the BET method. The textual studies were made using a sorptometer ASAP 2020 made by Micromeritics.

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**Notes**

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