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Utilization of Lanthanum-Modified Seed Crystals Produced Via Microbiological Production for the Purification of Calcium and Phosphorus from Household Wastewater

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Abstract: The synthesis of lanthanum-modified seed crystals via microbial mediation and their subsequent application in removing phosphorus and calcium from domestic wastewater is the focus of this study. Through the action of microorganisms, particularly Pseudomonas aeruginosa, lanthanum is incorporated into the seed crystals, enhancing their performance in wastewater treatment. The research demonstrates notable success in utilizing these modified seed crystals for wastewater treatment, with impressive removal efficiencies observed for both phosphorus and calcium. After multiple applications, significant reductions were achieved, with phosphorus removal reaching 94% and calcium removal at 60%. This study delves into the underlying mechanisms driving this enhanced performance, identifying key processes such as chemisorption, intra-particle diffusion, electrostatic contact, ligand exchange, and induced crystallization. Understanding these mechanisms is crucial for optimizing the design and operation of wastewater treatment systems utilizing these modified seed crystals. These experiments validate the efficacy of the modified seed crystals across different scenarios, particularly highlighting their superior performance in phosphorus removal, with an efficiency of 95%. Given the current environmental challenges posed by wastewater pollution and the increasing demand for sustainable treatment technologies, the development of lanthanum-modified seed crystals holds significant promise. Their application in domestic wastewater treatment presents a viable and effective solution, offering a pathway towards cleaner and healthier water resources. Its appeal extends to both developed and developing nations, offering flexibility to adapt to local needs by integrating it with conventional (above-ground) natural or engineered water and wastewater treatment technologies.

Keywords: *lanthanum; wastewater; Calcium and Phosphorus; Characterization; Modified Seed Crystals;*

1. Introduction

In recent years, the contamination of domestic wastewater with phosphorus (P) and calcium (Ca2+) has garnered significant attention due to its detrimental effects on the water cycle and aquatic ecosystems [1]. The enrichment of phosphorus in water bodies fosters aggressive algae growth, depleting dissolved oxygen levels and endangering the survival of fish and other aquatic organisms, thereby deteriorating water quality [2], [3], [4]. Additionally, an excess of calcium ions can lead to sludge disintegration and contribute to hardness overload in sewage reuse, exacerbating environmental concerns [5], [6], [7]. Therefore, the removal of phosphorus and calcium from sewage is crucial for mitigating eutrophication and enhancing water quality. Moreover, phosphorus is a vital fertilizer essential for food production, emphasizing the importance of its efficient recovery for resource reuse [8]. Various methods, including chemical precipitation, biological processes, and adsorption, are currently employed for phosphorus removal from sewage [9], [10]. Chemical precipitation, utilizing soluble metal salts like iron and calcium ions to form insoluble precipitates with phosphate, generates substantial chemical sludge during the removal process [11]. While biological methods are cost-effective, they often face challenges such as fluctuations in influent composition and a shortage of on-site carbon sources [12], [13]. Consequently, adsorption technology is gaining popularity due to its environmental friendliness, ease of operation, and economic benefits [14].

To address the need for efficient, environmentally friendly adsorbents, bio-synthetic materials have garnered attention, with microbially induced calcium precipitation (MICP) emerging as a promising option [15]. MICP, primarily utilized for water and soil remediation, possesses the unique ability to induce crystallization [16], [17]. Two main crystallization techniques, namely the Calcium phosphorus (Ca-P) and struvite processes, have been developed for phosphorus removal. While the struvite technique requires a constant supply of magnesium sources, increasing operational costs, the

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prevalence of high calcium content in wastewater favors the application of the Ca-P process [12], [18]. Additionally, the use of isomorphous seed crystals (SC) in MICP facilitates crystallization, enhancing phosphorus and calcium removal [20], [21]. In this study, lanthanum (La) was incorporated into seed crystals (SC) through a Pseudomonas-mediated method to enhance adsorption performance [22], [23]. La, known for its strong affinity for phosphorus, was loaded inside and on the surface of MICP to form modified seed crystals (MSC), thereby improving ligand exchange capacity and chemical stability. The presence of MSC reduced the activation energy barrier for crystallization product formation and altered the solid-liquid equilibrium, thermodynamic parameters, and energy of the system [24]. Batch experiments were conducted to optimize MSC preparation and assess phosphorus reduction using various kinetic models. he removal mechanism was elucidated through scanning electron microscopy-energy disperse X-ray (SEM-EDX), Fourier transforms infrared spectroscopy (FTIR), X-ray diffraction (XRD), and Zeta potential analysis. Furthermore, the adaptability and stability of MSC in simulated wastewater with varying water quality parameters were examined and analyzed. In arid and semi-arid regions where groundwater resources are overexploited, MICP technology serves as a valuable tool to supplement water supply. Its appeal extends to both developed and developing nations owing to its durability, capacity to remove diverse pollutants, environmental friendliness, and reduced reliance on energy and chemicals. As an integral component of integrated water resources management, MICP implementation ensures the sustainability of groundwater and surface water sources. Furthermore, MICP presents innovative solutions for safe wastewater reclamation and reuse, including indirect potable reuse facilitated by environmental buffers and the establishment of a psychological barrier.

1.1 MICP System

MICP, or microbially induced calcium precipitation, is a biologically mediated process that involves the precipitation of calcium carbonate minerals through microbial activity. This process is widely studied and applied in various fields, including environmental remediation, construction, and geotechnical engineering. In the MICP system, specific types of bacteria, such as ureolytic bacteria, play a crucial role. These bacteria produce urease enzymes that catalyze the hydrolysis of urea into ammonia and bicarbonate ions. The increase in pH resulting from urea hydrolysis promotes the precipitation of calcium carbonate (CaCO3) from calcium ions present in the surrounding environment.

- MICP can be employed to improve the mechanical properties of soils by cementing soil particles together through calcium carbonate precipitation. This technique is particularly useful for stabilizing loose or sandy soils, reducing soil erosion, and enhancing the load-bearing capacity of foundations.
- MICP can facilitate the immobilization or precipitation of heavy metals and other contaminants in groundwater through the formation of insoluble carbonate minerals. This helps in reducing the mobility and bioavailability of contaminants, thereby mitigating environmental risks.
- MICP has been explored as a potential method for self-healing concrete. By incorporating ureolytic bacteria and urea into concrete mixtures, cracks that form over time can be healed through the precipitation of calcium carbonate, preventing further deterioration and extending the lifespan of concrete structures.
- MICP can be harnessed for the production of bio-based construction materials, such as biocement and biostone. These materials are eco-friendly alternatives to traditional cementitious materials and can be produced using low-energy and low-cost processes.

2 Removal mechanism and methodology

The implementation of MICP (microbially induced calcium precipitation) worldwide has been notable, especially in utilizing primary, secondary, and tertiary effluents from wastewater treatment. These effluents serve as a valuable resource for MICP systems, contributing to contaminant attenuation through various mechanisms as they pass through different zones within the system. Figure 1 outlines the MICP methodology, illustrating how filtration, biodegradation, chemical precipitation, adsorption, ion exchange, and dilution mechanisms work together to remove contaminants. Among these mechanisms, filtration, biodegradation, and adsorption stand out as primary removal processes. Filtration involves the physical removal of suspended particles and colloids from the wastewater as it passes through porous media or filtration beds. Biodegradation relies on microbial activity to break down organic contaminants into simpler, less harmful substances. Adsorption involves the adhesion of contaminants onto the surface of solid particles or media, effectively removing them from the water phase. For long-term sustainability and effectiveness, it is essential to design and operate MICP systems with a focus on removing biodegradable contaminants. By prioritizing the removal of these contaminants, MICP systems can contribute to environmental protection and ensure the continued viability of the system over time.

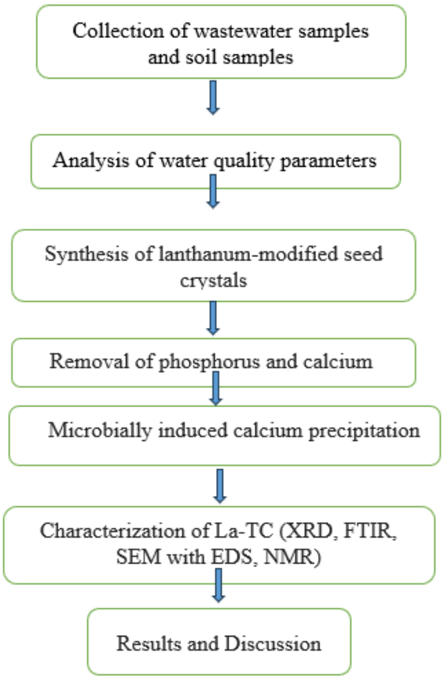

Figure 1 Research Methodology

Several factors significantly influence the removal efficiencies of MICP (microbially induced calcium precipitation) systems. These include source water quality, site-specific hydrogeological conditions, the type of MICP system employed, and the applied process conditions. The overarching goal of water quality improvement within MICP systems is the elimination of suspended solids and microorganisms. However, additional benefits include the reduction of dissolved organic carbon (DOC) concentrations and the removal of nitrogen species through biological processes. During soil aquifer treatment within MICP systems, both nitrification and denitrification processes can occur. The effectiveness of nitrogen removal hinges on several factors, including residence time, the ratio of DOC to nitrogen, and prevailing redox conditions. Optimizing these parameters is essential for maximizing nitrogen removal efficiency within MICP systems and achieving desired water quality outcomes.

2.1 Site selection and sample collections

The choice of site is crucial to a MICP system's effectiveness. When evaluating and choosing a site, the following variables are crucial: (i) soil depth; (ii) soil penetration; and (iii) depth to groundwater (depth of vadose zone). (iv) the thickness of the aquifer (depth from the foundation to water table). Soils that are sufficiently permeable to provide high level of infiltration are the ideal locations for MICP systems' infiltration river basins. But as the effluent seeps through, the soils must also be fine enough to offer effective filtration as well as enhancement. The experimental set up was carried out in Civil Engineering Department, Sathyabama Institute of science and technology, Chennai during Feb-March 2020. The geographic location of the study area is 12°52′23″N, 80°13′19″E in Figure 2.

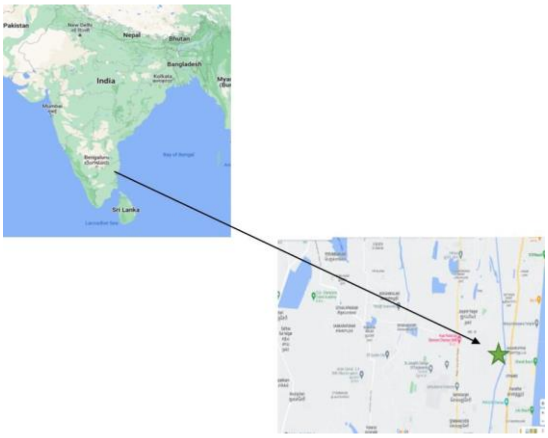

Figure 2 Location of study area

Soil classification study conducted at Sathyabama campus in Chennai, where soil samples were collected from 10 different locations at depths ranging from 0.0 to 1 meter (referred to as topsoil). These locations include Block 12, Field lab, Near Central Library, Bio Department, Research Centre, Front Side, Back Side of Architectural Department, Admin Block, Back Side, Near Ladies' Hostel, Boys’ Hostel, and near Jeppiar sewage treatment plant (JSTP). The soil samples were analyzed based on their composition, specifically focusing on the percentage of loamy sand, fine sand, and clayey sand. The classification was done according to the Indian Standard System of Soil classification.

2.2 Fabricated Soil Columns

Before conducting the column study, batch studies were conducted to assess the vadose zone's capacity to transform sewage treatment wastewater (STWW) and to investigate the MICP parameters responsible for reducing wastewater elements. The batch study involved two methods: pre- and post-soil filling [3]. In the pre-soil filling method, a bucket with a height of 20 cm was used, with holes made at the bottom. These holes were covered with fine mesh to collect water without soil particles. Gravel and pebbles were then added on top of the mesh up to a depth of 2.5 cm. After filling the soil, it was properly sieved through a 2 mm sieve before placing it over the gravel and pebbles to a depth of 12 cm. Once the water was poured, the ponding depth was maintained at 5 cm with a 2 cm freeboard. The STWW collected from Sathyabama Jeppiar sewage treatment plant (STP) was poured to a depth of 5 cm for the batch study. During the experiment, the soil column was kept fully saturated. As the water penetrated the soil, it was collected at the bottom of the bucket as effluent. The collected effluent was then analyzed to compare its characteristics with the influent. In the column operations, a one-day wetting followed by a one-day drying process was initially conducted. During the drying process, the soil was spread on exposed earth to enhance it with oxygen and microorganisms from the environment. After drying, the soil was returned to the column and subjected to STWW. The batch setup is illustrated in Figure 3.

Figure 3 Fabricated Soil Columns

2.3 Characterization Techniques

Characterization of MICP can be conducted using various analytical techniques to understand the physical, chemical, and structural properties of the soil and its interactions with wastewater. Three commonly used techniques for characterization are Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and Nuclear Magnetic Resonance (NMR) spectroscopy.

- FTIR spectroscopy is a powerful tool for analyzing the functional groups present in soil samples. It provides information about the chemical composition and structure of organic and inorganic compounds in the soil. FTIR can be used to identify specific functional groups such as hydroxyl, carbonyl, and amine groups, which are important in understanding soil organic matter and its interaction with contaminants in wastewater.
- SEM is a microscopy technique that provides high-resolution images of the surface morphology and microstructure of soil particles. It allows for the visualization of soil aggregates, pore structures, mineral compositions, and the distribution of organic and inorganic components. SEM can provide valuable insights into the physical characteristics of soil samples and their interaction with contaminants and microbial communities.
- NMR spectroscopy is a non-destructive technique that provides information about the molecular structure and dynamics of soil organic matter. It can be

used to characterize the composition, molecular weight distribution, and mobility of organic compounds in soil samples. NMR spectroscopy can also provide insights into the interactions between soil organic matter and water molecules, which are important in understanding the transport and fate of contaminants in SAT systems.

By combining these techniques, researchers can gain a comprehensive understanding of the properties and behavior of soil in SAT systems, which is essential for optimizing treatment efficiency and ensuring the sustainable management of wastewater resources.

3. Results and Discussion

3.1 Physical characteristics

The study focused on assessing the efficacy of MICP (microbially induced calcium precipitation) in removing physical and chemical pollutants from wastewater. Initially, various parameters characterizing wastewater quality were monitored, and the most significant parameters for assessing the degree of quality improvement were selected for continuous monitoring throughout the study. The process involved subjecting soil columns to wetting and drying cycles to simulate the MICP process. Specifically, one-day wetting followed by one-day drying cycles were employed. The initial pH of the sewage treatment plant wastewater (STWW) influent was measured. PH values for the effluent water after passing through three different soil types. It was observed that the average pH of the effluent water was lower than that of the influent, indicating an overall increase in pH values. This increase could be attributed to the production of CO2 during organic biodegradation processes. Additionally, turbidity, caused by suspended solids, was monitored throughout the study. The initial turbidity level of the STWW before soil aquifer treatment (SAT) was recorded at 7.24 NTU. Tracking changes in turbidity provides insights into the effectiveness of the MICP process in removing suspended solids and improving water clarity. Continuously monitoring these parameters allows for a comprehensive assessment of the performance of MICP systems in treating wastewater and improving water quality.

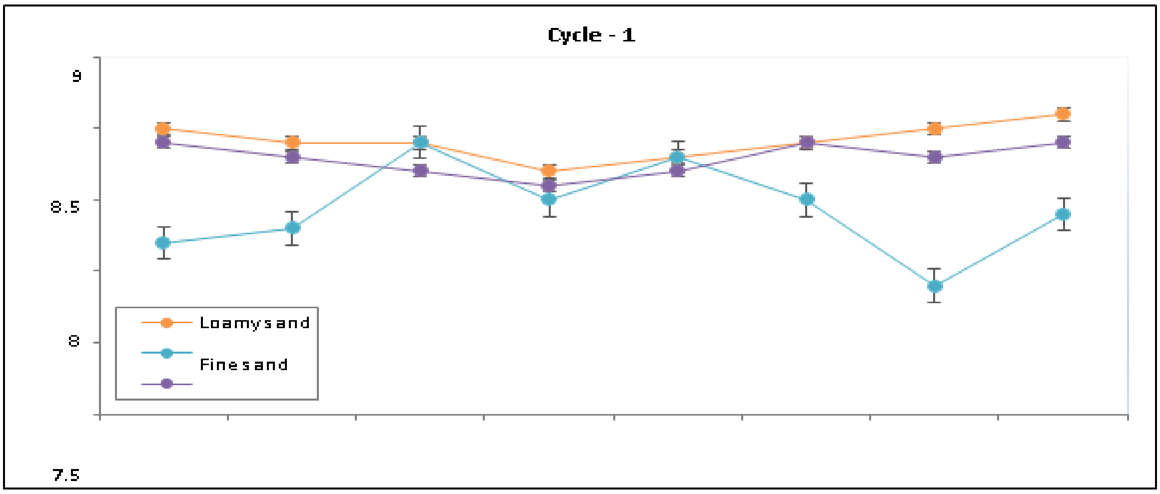

Figure 4 PH variations in soil columns in two-day wetting/drying cycle

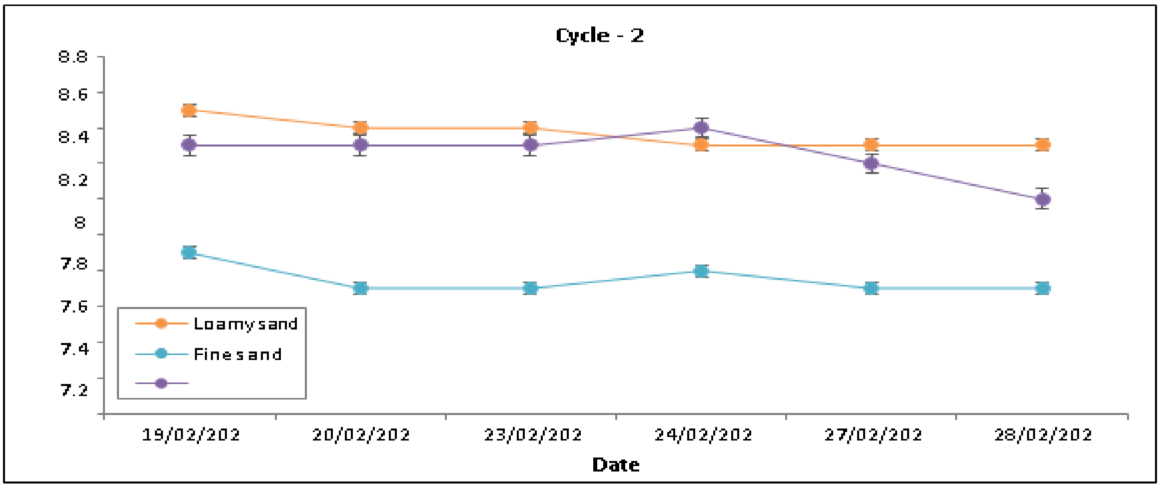

Figure 5 PH variations in soil columns in one day wetting /drying cycle

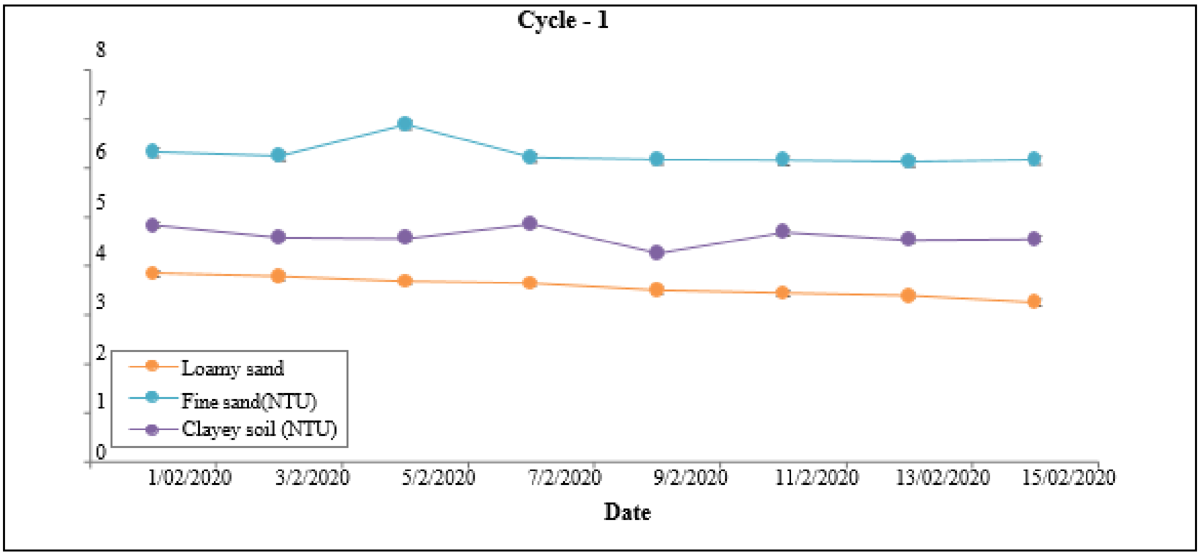

Figure 6 Turbidity variations in soil columns in one day wetting /drying cycle

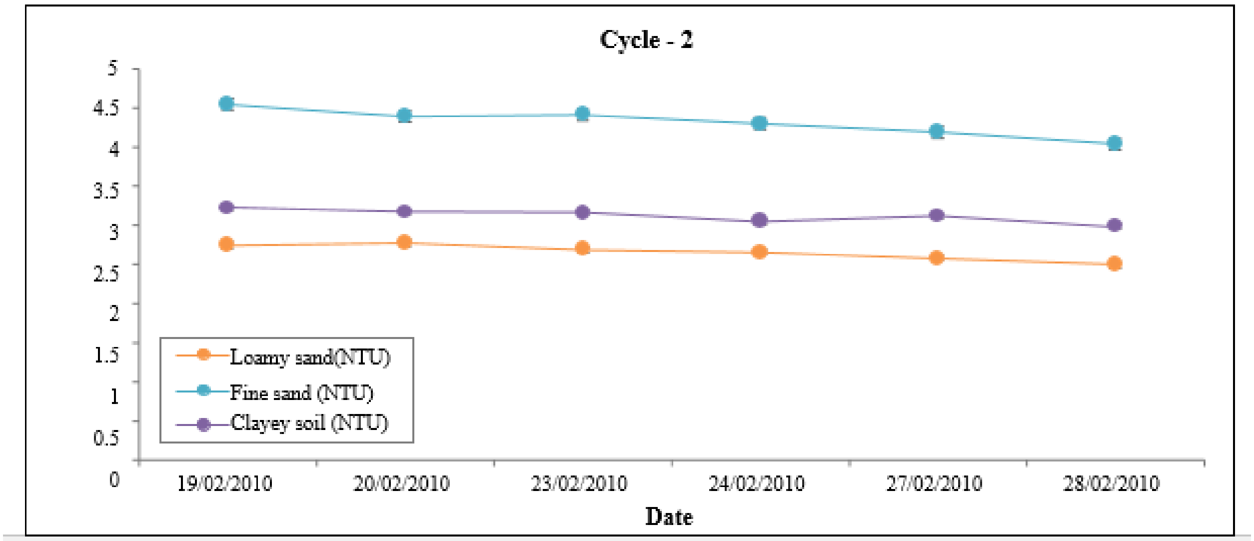

Figure 7 Turbidity variations in soil columns in two days wetting /drying cycle

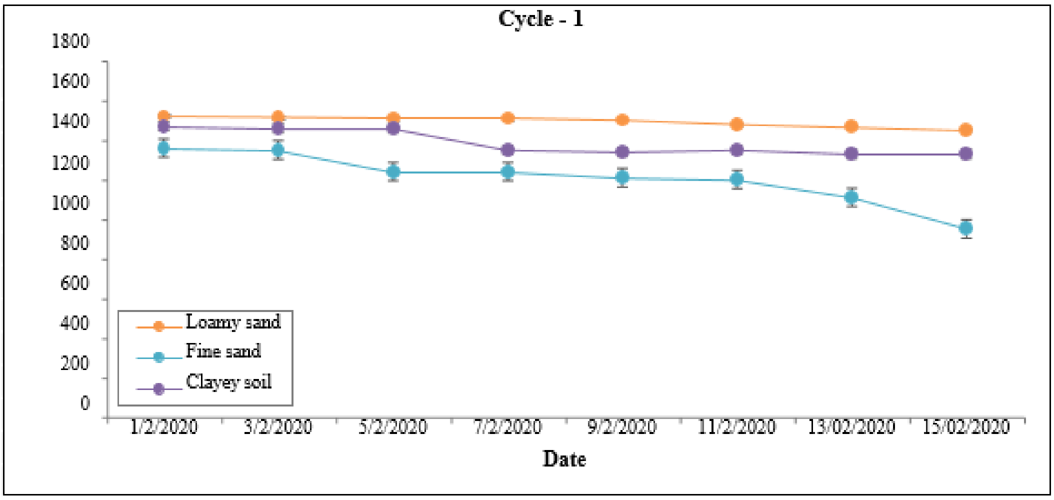

Figure 8 TDS variations in soil columns in one day wetting/drying cycle

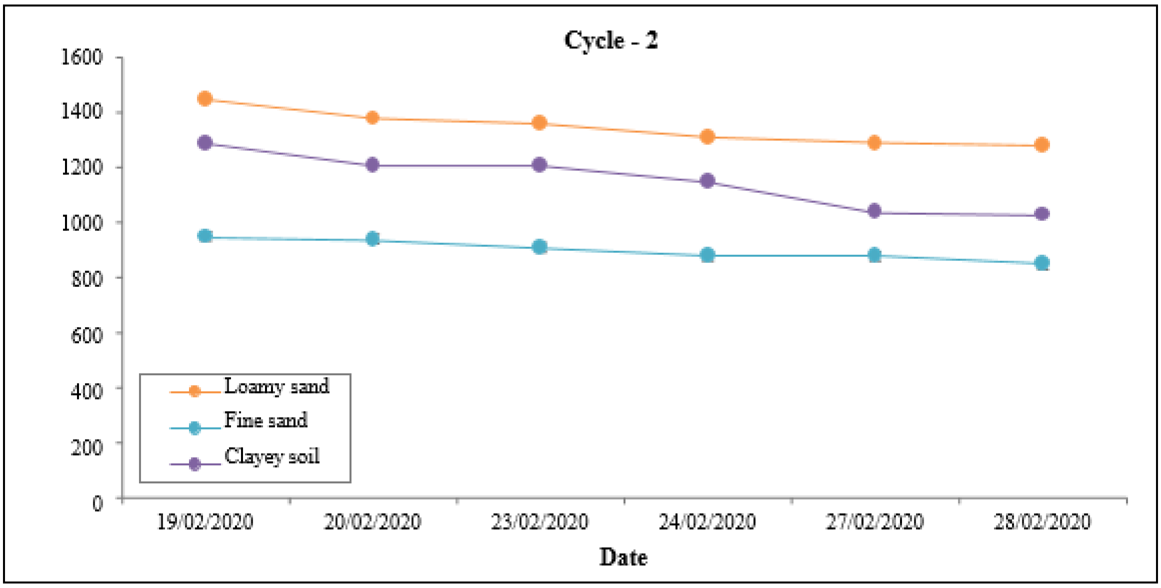

Figure 9 TDS variations in soil columns in two days wetting/drying cycle

Figure 7 and 8 illustrate the turbidity values for the three types of soil samples after MICP treatment. Interestingly, it was observed that loamy sand soil exhibited superior turbidity removal efficiency under certain conditions [8]. Furthermore, Total Dissolved Solids (TDS) levels were monitored throughout the study, with an initial level in the sewage treatment plant wastewater (STWW) measured at 1580 mg/l. Table 5 presents the efficiency of TDS removal for the three types of soil after MICP treatment. Remarkably, fine sand demonstrated better TDS removal efficiency under specific wetting/drying cycles. The effectiveness of MICP as a method for removing contaminants and improving water quality. MICP holds promise for various applications, particularly in groundwater recharge and wastewater reuse initiatives, where stringent water quality standards must be met. By continually refining MICP processes and optimizing operational parameters, its potential for sustainable water treatment solutions can be further realized.

3.2 CHARACTERISICS OF SOIL BEFORE AND AFTER MICP

The soil was characterized to determine the chemical pollutants that has adsorbed in the soil after MICP and also to determine which soil will be suitable in removing the chemical pollutants. Soil samples were extracted from the columns to determine the chemical pollutants like Arsenic (As), Sodium (Na), Magnesium (Mg), Iron (Fe) and Zinc (Zn).

3.2.1 Adsorption isotherms

Phosphate adsorption isotherms are graphical representations of the relationship between the concentration of phosphate ions in a solution and the amount of phosphate adsorbed onto a solid adsorbent at equilibrium. These isotherms provide valuable information about the adsorption capacity, affinity, and mechanisms of phosphate removal by the adsorbent material. The Langmuir isotherm assumes a monolayer adsorption onto a homogeneous surface with a finite number of identical sites. It is described by the equation:

$$q = \frac{Q_{\max} K_L C}{1 + K_L C}$$

Where, q is the amount of phosphate adsorbed per unit mass of adsorbent (mg/g).

C is the equilibrium concentration of phosphate in solution (mg/L).

Qmax is the maximum adsorption capacity of the adsorbent (mg/g).

K_L is the Langmuir adsorption equilibrium constant (L/mg).

The Freundlich isotherm describes heterogeneous adsorption onto a surface with multiple sites of varying affinities. It is represented by the equation:

$$q = K_F C^{\frac{1}{n}}$$

Where, q is the amount of phosphate adsorbed per unit mass of adsorbent (mg/g).

C is the equilibrium concentration of phosphate in solution (mg/L).

KF and n are Freundlich constants related to adsorption capacity and intensity, respectively.

The Temkin isotherm considers interactions between adsorbate and adsorbent, incorporating a linear decrease in adsorption energy with surface coverage. It is expressed as:

$$q = B\ln(AC)$$

Where, q is the amount of phosphate adsorbed per unit mass of adsorbent (mg/g).

C is the equilibrium concentration of phosphate in solution (mg/L).

A and B are Temkin isotherm constants.

These isotherms are commonly used to analyze experimental data from batch adsorption experiments and determine the adsorption capacity and affinity of various adsorbents for phosphate ions. By fitting experimental data to these models, researchers can gain insights into the mechanisms and kinetics of phosphate adsorption onto different materials, aiding in the design and optimization of phosphate removal processes

3.2.2 FTIR Analysis of La-TC

The FT-IR spectra primarily reflect changes in functional groups. Comparing the FT-IR spectra of TC and La-TC, several distinctive bands were observed. The band appearing at 3609 cm−1 was attributed to the hydroxyl group of Lanthanum hydroxide, while the peak at 650 cm−1 was assigned to LaO. Across all samples, the characteristic peak near 3425 cm−1 corresponded to the OH stretching vibration peak of surface-adsorbed water molecules. Notably, a sharp absorption peak at 1383 cm−1 was observed on the surface of La-TC, attributed to the vibration pattern of anions of La(NO3)3 embedded into the structure of TC. Comparatively, in La-TC-P, the feature peaks might be related to the bend vibration of O PO at the wavenumber of 614 cm−1, indicating the formation of an inner-sphere complex during the phosphorus adsorption process. Additionally, a strong feature peak at 1053 cm−1 was attributed to the asymmetric vibration of the P O bond in the PO3- 4 group on the surface of La-TC-P.

On the surface of La-TC, a sharp peak at about 1500 cm−1 was observed, belonging to the asymmetric stretching of CO2- 3, possibly caused by CO2 absorbed onto the surface of La(OH)3. The spectrum of La-TC-P-D was relatively consistent with that of La-TC. FTIR spectroscopy was conducted using a Nicolet model 510P Fourier transform infrared spectrometer equipped with Nicolet EZ Omnic E.S.P. software, Version 4.1a. The analysis was performed on organic isolates using the pressed-pellet method. Before analysis, the isolates were dried at 65°C for 24 hours to remove any residual moisture. Pellets for analysis were prepared by combining 1 mg of dried isolate with 100 mg of optical-grade potassium bromide (KBr). The mixture was then ground using a mortar and pestle and pressed into a pellet using a screw press. The sample was allowed to sit for at least 15 minutes, allowing the KBr to fuse, prior to analysis. To ensure accurate analysis, a background spectrum of CO_2 was generated at the beginning of each analysis session [12-15]. This background spectrum was automatically subtracted from each sample spectrum to eliminate any interference. Thirty-two scan spectra were obtained across a range of wave numbers from 4000 to 400 cm^−1

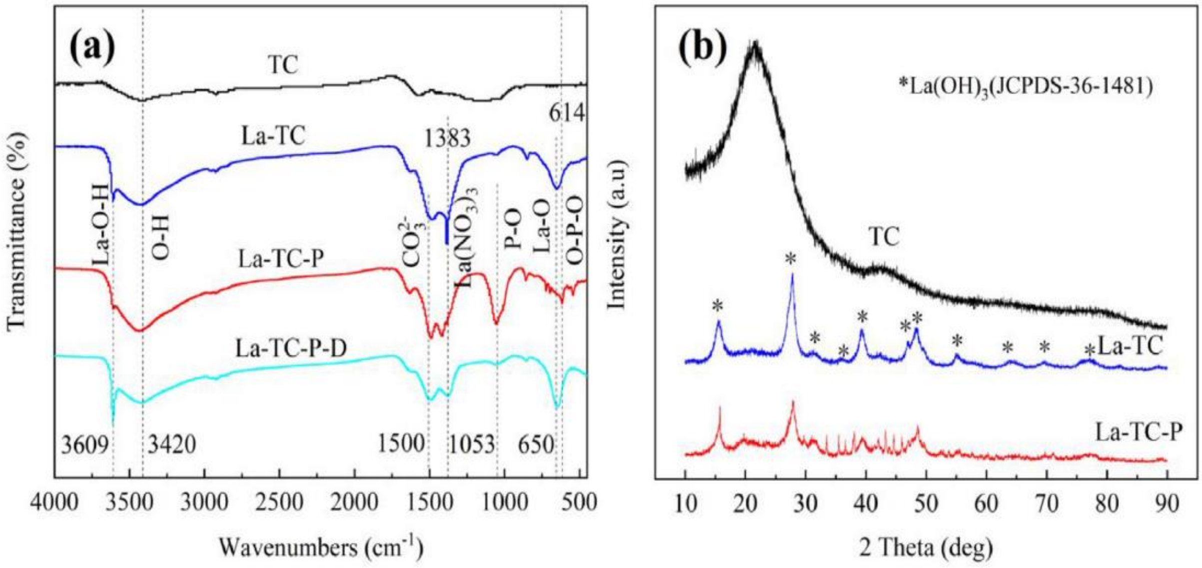

Figure 11 (a) FTIR analysis of La-TC, (b) XRD patterns of La-TC

3.2.3 SEM Analysis of La-TC

The SEM images provided insights into the morphological changes of TC, La-TC, and La-TC-P, while the EDS element diagram of La-TC-P offered information on elemental distribution. The SEM image of TC (Figure 1a) revealed a relatively smooth and clean surface, with enlarged pores resulting from KOH activation. In contrast, the surface of La-TC exhibited a rough, short rod-like structure, indicating successful lanthanum loading onto the TC surface. Following phosphate absorption, cobweb-like substances were observed hanging on the rod-like particles of La-TC-P. The EDS spectrograms illustrated well-dispersed lanthanum distribution on La-TC, facilitating efficient phosphate adsorption. Additionally, uniform phosphorus adsorption on the surface of La-TC-P was evident.

The SEM photographs (Figure 12) revealed distinct surface characteristics among the biosorbents, providing crucial insights into their textural structure, pore distribution, and surface heterogeneity, which directly influence their adsorption properties. Surface roughness, characterized by fractal dimension, and porosity, represented by lacunarity, are important parameters related to the periodic arrangement of cellulose, hemicellulose, and lignin in the biosorbents [16-20]. Among the five feedstocks studied, eucalyptus bark (EB) exhibited the highest values for fractal dimension (DF = 1.732) and lacunarity (0.613), indicating a greater surface roughness. This suggests that EB possesses a more irregular and rough surface texture compared to the other biosorbents. While there wasn't much variation in the fractal dimension and lacunarity among the other four biosorbents, rice straw (RS) demonstrated the lowest values for both parameters. This indicates that RS has a relatively smoother surface texture compared to the other biosorbents studied.

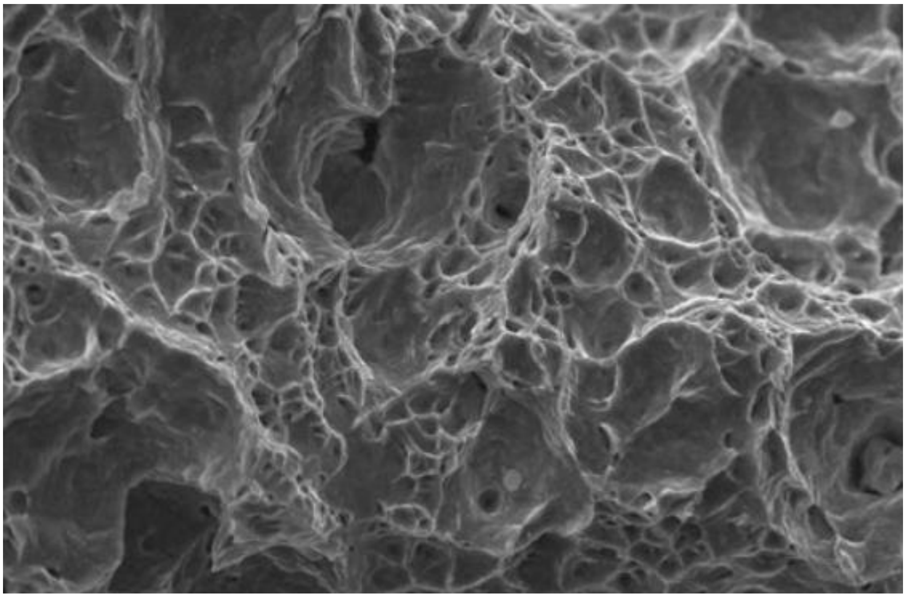

Figure 12 SEM Analysis of La-TC

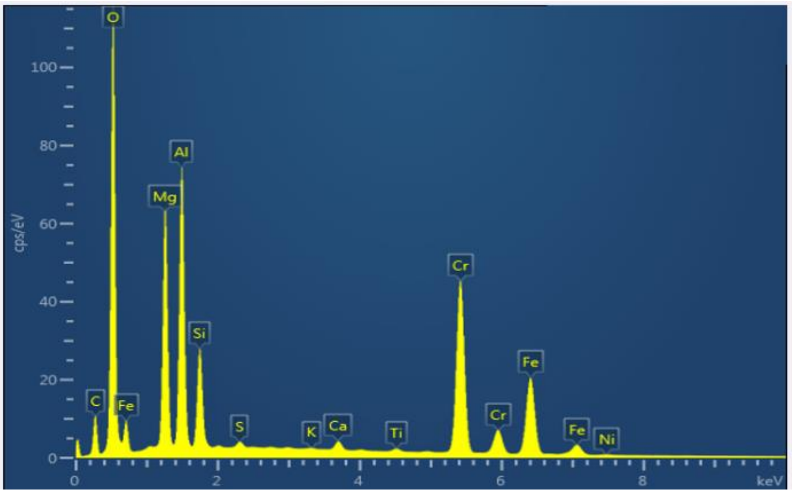

Figure 12a EDS Analysis of La-TC

3.2.4 NMR Analysis of La-TC

MR (Nuclear Magnetic Resonance) analysis of biosorbents involves using NMR spectroscopy techniques to investigate the molecular structure and composition of the biosorbent material in Figure 13. The NMR data are interpreted to gain insights into the molecular structure, composition, and functional groups present in the biosorbent material [21,22].This information can help in understanding the mechanisms of sorption, interactions with adsorbates, and potential applications of the biosorbent in environmental remediation, wastewater treatment, and other fields.

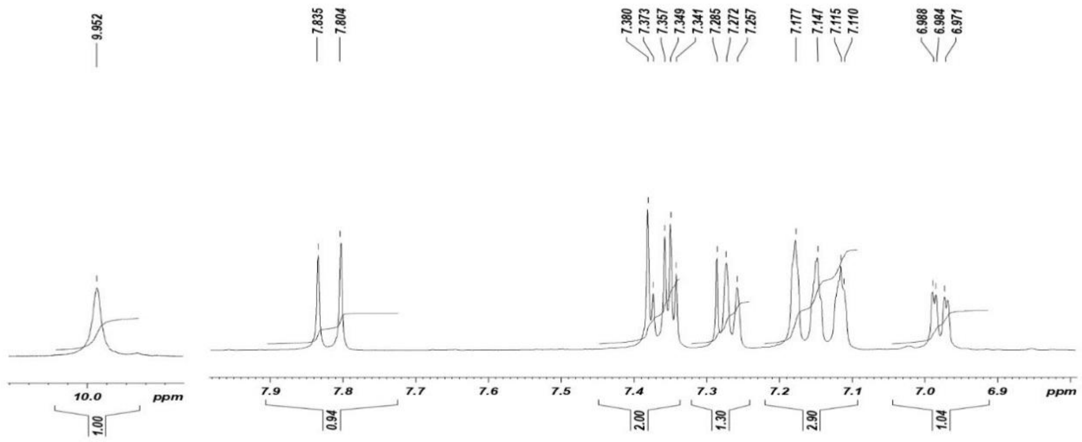

Figure 13 NMR Analysis of bio sorbent

Conclusion

In this study, biochar derived from eco-friendly and economically viable platanus ball fiber was utilized for the first time. Through modification with lanthanum nitrate, the resulting adsorbent, La-TC, demonstrated exceptional performance in removing and recovering phosphate from real wastewater. La-TC exhibited a remarkable saturated adsorption capacity for phosphate, reaching as high as 150mg/g, surpassing many other biochar-based adsorbents available. Additionally, La-TC displayed effective phosphate adsorption across a wide pH range of 5, along with high selectivity, stability, and regeneration capability.

Adsorption kinetics studies revealed that the adsorption behavior of La-TC followed the Langmuir model provided an excellent description of the adsorption isotherm. In summary, La-TC emerged as a promising candidate for phosphate adsorption, offering efficient removal and potential for phosphate reuse from real wastewater sources. This study highlights the significant potential of La-TC as a sustainable and effective solution for addressing phosphate pollution in water environments.

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