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Vivianite: A sustainable route for phosphorus recovery from waste water and sewage sludge

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Abstract
Recovery of phosphorus (P) from sewage sludge and wastewater has emerged as a priority activity with rising demand for P resources and the necessity for efficient waste treatment. In recent years, conversion of phosphorus to vivianite ($Fe_3(PO_4)_2 \cdot 8H_2O$), a valuable and useful mineral, has gained increased attention. Vivianite recovery is promising due to the fact that the process is easy to control, has high phosphorus recovery efficiency and the product has numerous practical applications. This review initially examines the techniques employed for detection and quantification of vivianite content, crucial in assessing the effectiveness of the recovery process. It secondly briefly outlines advanced technologies applied for vivianite formation. These include anaerobic fermentation, chemical precipitation, electrochemical crystallization, bio mineralization and anaerobic digestion. Furthermore, the review considers significant factors that affect the formation of vivianite, including pH value, iron-to-phosphorus (Fe/P) molar ratio, concentration of sulfate, microbial activity, supersaturation degree, organic content and seed crystal usage. The review is generally facilitating the production of cleaner and more effective technologies with the ability to recover phosphorus and convert waste into useful resources, hence ensuring sustainable wastewater treatment and circular economy.

Keywords: Phosphorus (P) recovery, vivianite ($Fe_3(PO_4)_2 \cdot 8H_2O$), vivianite formation

Introduction

Phosphorus (P) is a vital nutrient needed for every life form, particularly in plants. It is an essential element for various physiological processes, such as energy transfer (ATP), photosynthesis, root growth, seed development, stem growth and imparting resistance to root-disease (Liu *et al*., 2013) [16]. Due to its importance in agriculture, phosphorus is an integral part of synthetic fertilizers used to fuel world food supplies. Rock phosphate is now the major supply of phosphorus fertilizers, but it is a finite, non-renewable resource (Cordell *et al*., 2009) [6]. Isaac Asimov already, in the 1970s, cautioned that phosphorus was "life's bottleneck," pointing out that, in contrast to other resources energy or protein phosphorus has neither a synthetic alternative nor substitute.

The world population is likely to grow to more than 9 billion by 2050, and food demand is likely to shortage. This will put even more pressure on agri-systems to yield more food with phosphorus-based fertilizers. Presently, the total yearly global rock phosphate consumption is approximately 1 million tons, and it will increase significantly perhaps up to 70 million tons by 2050. If trends persist, it is estimated that half of the economically extractable rock phosphate of the world might be exhausted in the next 60-70 years. Additionally, the geographically skewed distribution of these reserves, being mainly located in a limited number of countries, puts future access, affordability, and geopolitical hazard at risk. Hence, there is an imperative to tap sustainable phosphorus recovery and recycling options especially from secondary sources like wastewater, farm runoff and sewage sludge to guarantee long-term food security and facilitate a circular economy.

Phosphorus is a non-sustitutable plant macronutrient, a fertilizer and food supply bottleneck, thus its demand is increasing worldwide. Since it is the second most soil-imporvised after nitrogen, it has to be supplemented externally. The active forms of P impacted the environment. Precipitated waste water as vivianite needs to be utilized in order to produce an economic fertilizer that addresses this problem without damaging the environment.

Waste water treatment and management are in imbalance with waste water production. The major site of phosphorus convergence is waste water plants.

1.3 mt of phosphorus was removed from wastewater annually. 15-25% of the total world needs of phosphorus could be met by the recovered phosphorus from waste water (Venkiteshwaran *et al*., 2018) [32]. Eutrophication is caused by water that is rich in P. To minimize pressure on the demand for rock phosphate and to stop eutrophication, recovery of P from waste water is important. Vivianite precipitation, hydroxyl apatite formation and crystallization of struvite are now some of the common techniques used in recovering P from wastewater (Sun *et al*., 2020) [31]. Scholars have been interested in P recovery as vivianite due to its economic value, easy availability, and natural omnipresence (Prot *et al*., 2020) [24].

Vivianite formation

Vivianite, as per Schoepfer *et al*. (2019) [29], is an iron phosphate mineral that is commonly found in soil or lake sediments in anaerobic and Fe-enriched environments. Pure vivianite, as described by Hearn *et al*. (1983) [9], is white and transparent when first formed but becomes opaque and green or blue when oxidized in air. Also, bogs, hydrothermal deposits, and root systems have been discovered with vivianite (Dijkstra *et al*., 2016) [7]. Vivianite would convert to metavivianite, if Fe (III) was more concentrated.

Vivianite needs both iron and phosphorus. Iron dosage is assisted with phosphate flocculation. Wastewater consists of high concentrations of phosphorus in three forms: polyphosphates, inorganic phosphorus, and organic phosphorus. Iron ions (Fe^{3+}) are reduced to Fe^{2+} by dissimilatory metal-reducing bacteria (DMRB), while organic phosphorus is reduced to phosphate by anaerobic microorganisms. Fe^{2+} and PO_4^{3-} concentrations rise in the local environment that is created while these two microbial processes persist (Wu *et al*., 2021; Zhang *et al*., 2021) [30, 46].

$$\text{Organic Phosphorus} \longrightarrow PO^{-3}{}_4$$

$$Fe(OH)_3 + 3H^+ + e^- \longrightarrow Fe^{+2} + 3H_2O$$

$$3\,Fe^{+2} + 2PO^{-3}{}_3 + 8H_2O \longrightarrow Fe_3(PO_4)_2.8H_2O$$

Vivianite has a number of benefits over struvite, including the following: 1) Vivianite's market price (10,000 €/ton) is significantly higher than that of MAP (500 €/ton) (Zhang *et al*., 2022) [47]; a good adsorbent for the removal of heavy metals such as arsenic (Cai *et al*., 2020) [3], and a slow-release fertilizer that provides crops with a constant source of nutrients (Jowett *et al*., 2018) [12]. Vivianite also possesses the operational advantage of precipitating at a relatively moderate pH range of 6-8, which is favorable for most wastewater treatment plants and facilitates easier control of operations (Li *et al*., 2021) [14]. High recovery efficiency for phosphorus have also been exhibited by vivianite recovery processes; recovery efficiencies ranging from 82.6% have been reported in some researches (Cao *et al*., 2019) [4].

Vivianite detection

XRD was the only crystalline vivianite identified. Poorly crystallizing vivianite can be neglected (Salehin *et al*., 2020) [28]. Also, for assuring the three highest peaks would be easily detectable, a sample should consist of a minimum of 5% weight of vivianite (Rothe *et al*., 2014) [25]. Vivianite detected by XRD is also affected by several parameters like nodule size in the crystal, proportion of vivianite, and crystallinity. Therefore, qualitative research of vivianite is better than quantitative analysis. The functional groups of vivianite are often identified through Raman and IR-absorption spectroscopy. Two types of spectrums with comparable bands exist. Unfortunately, it seems that neither of them can identify Fe^{2+}-OH (Ogorodova *et al*., 2017) [22]. Mossbauer spectroscopy and XAS are two quantitative methods that are relatively reliable.

Different methods involved in vivianite recovery from waste water are

1. **Chemical method:** Crystallization, Adsorption and Use of iron hydroxides
2. **Biological method:** Different iron sources used for vivianite formation are Ferrihydrate, Ferric citrate, $FeCl_3$, $FeSO_4$ and Ferric citrate

1. Chemical Precipitation

Chemical Precipitation (CP) is the most common method of P recovery. Oversaturation, birth of crystals, expansion and agglomeration are the main stages of crystallization (Carrillo *et al*., 2020) [5]. Crystalline nuclei initially occur rapidly after supersaturation. The molecules then combine to develop an ordered structure. Finally, the precipitate sizes increase due to small crystals attaching to large/agglomerated crystals (Kataki *et al*., 2016) [13]. Phosphate precipitates are normally generated by dosing metal salts (Melia *et al*., 2017) [18]. Fluidized bed crystallizers (FBC) were employed to accelerate the synthesis of vivianite from CP (Priambodo *et al*., 2017) [23].

2. Electrochemical method

Phosphorus recovery from wastewater is a prevalent application for electrochemical (EC) methods (Carrillo *et al*., 2020) [5]. It involves applying electrochemical reactions to accumulate phosphate, from which the phosphate will be recovered through precipitation (Ye *et al*., 2019) [43]. Ion-exchange membranes allow for the effective isolation of the anode and cathode compartments to prevent the unwanted reaction of oxidation and reduction products (Ichihashi and Hirooka, 2012) [10]. Electrochemical systems may utilize zero-valent iron (ZVI) as the anode, thus a more cost-effective alternative compared to conventional chemical precipitation using ferrous or ferric salts (Wang *et al*., 2021) [34]. For effective recovery of phosphorus, ZVI at the anode is oxidized to form ferrous ions (Fe^{2+}), which migrate into the solution and react with phosphate to form vivianite.

3. Adsorption

Adsorbents like biochar, redmud, flyash were employed as adsorbents to reclaim vivianite from waste water. Where it enhances the iron reducing efficiency and enhances decomposition of complex organic matter inturn enhances the phosphorus release. It enhances the contact among iron and phosphorus by adsorption which may improve the P recovery.

4. Anaerobic digestion

Anaerobic digestion (AD) is an environmentally friendly and cost-effective way of sludge treatment (Yuan *et al*., 2019) [45]. $MgCl_2$ was added to precipitate along with phosphate and ammonia in the AD process to produce struvite, enhancing the dewaterability of the sludge (Melia *et al*., 2017) [18]. As vivianite attracts increasing scientific interest, $FeCl_3$ is increasingly being considered a better coagulant. With respect to P removal, volatile solids reduction, control of odor emission, and enhancement of sludge dewaterability, $FeCl_3$ was superior to $MgCl_2$ (Wilfert *et al*., 2018) [38]. Anaerobically, DIRB could possibly convert ferric ions to ferrous ions, which in turn would precipitate with phosphate to form vivianite. Interestingly, the most favorable pH range for methane synthesis (i.e., 6-8) was also the most favorable pH range for vivianite crystallization (Yang *et al*., 2015) [42].

5. Biological method

Under anaerobic conditions, for example, in wastewater and sludge, dissimilatory iron-reducing bacteria (DIRB) are typical bacteria that ferment ferric into ferrous ions (Wang *et al*., 2019) [33]. DIRB inoculation in sewage can stimulate the formation of biologically induced vivianite, which took over from chemical precipitation as the dominant crystallization mechanism (Wang *et al*., 2018) [35]. It must be mentioned that phosphate may be used for vivianite formation only when the P demand for DIRB has been entirely met. Conductive materials such as ZVI, magnetite, and graphite may significantly contribute to the reduction of ferric ions and vivianite biomineralization due to the electron transfer from DIRB to ferric ions (Shi *et al*., 2021) [30]. Vivianite also formed by utilization of dissimilatory iron reducing bacteria, where it utilizes organic carbon as an electron donor and ferric ion as electron acceptor results in reduction of ferric form of iron to ferrous form. Released ferrous ion combine with released phosphorus to form vivianite (Wang *et al*., 2022) [37].
Ex: *Geobacter sulfurreducens*

Recovering phosphorus (P) from wastewater, highlighting each of their advantages, disadvantages and capacity for recovery

1. **Chemical precipitation/crystallization:** Its disadvantages are the costliness of chemicals and the difficulty of effectively gathering up phosphorus. Its P recovery capacity is thus considered low.
2. **Adsorption:** Is known to be cheap and easy to implement. Despite these benefits, wastewater is needed to be pretreated, and the presence of competing ions will have some effect. It does, however, offer a high recovery potential for phosphorus.
3. **Biological treatment:** Or rather improved biological phosphorus removal (EBPR), can remove other contaminants like nitrogen, and it is economical with minimal chemical utilization. Nevertheless, it calls for stringent working conditions. However, it is chemically sensitive to limit microbial action and needs stringent operating conditions. It possesses a medium phosphorus recovery potential rating.
4. **Membrane processes:** Help in increased resource and energy recovery and provide excellent phosphorus removal efficiency. High operating, energy, and capital expenses are the primary detriments. Membrane techniques have a high potential for phosphorus recovery despite these hindrances.

Key parameters affecting vivianite production

The formation of vivianite ($Fe_3(PO_4)_2 \cdot 8H_2O$) is regulated by a variety of operating and environmental conditions

1. **pH:** The optimal range of pH for precipitation of vivianite has been reported by Wilfert *et al.* (2015) [39] as 6.0-8.0, in which solubility, ionic strength, and saturation index (SI) all form crystals. Acidic conditions enhance vivianite solubility in addition to the ferric iron reduction. However, alkaline conditions enhance the availability of phosphate but may favor the formation of iron hydroxide ($Fe(OH)_2$) rather than vivianite (Liu *et al.,* 2018; Cao *et al*., 2019) [15, 4]. Phosphorus recovery in electrochemical processes is optimal at pH 6-7 (Martin *et al*., 2020; Irdemez *et al*., 2006) [17, 11].
2. **Fe/P molar ratio:** To take into account side reactions like the formation of $Fe(OH)_2$, a good operating range of 1.5-2.0 is commonly recommended, though the theoretical stoichiometric ratio is 1.5. Nonetheless, elevated phosphate content can diminish iron requirements, and a ratio of 1.0 to 1.3 has been found in some studies to yield purer vivianite (Wang *et al*., 2018; Martin *et al*., 2020) [35, 17].
3. **Sulfide ions:** Theses generated from sulfate compete with phosphate for Fe^{2+}, amounts above certain thresholds are able to precipitate iron sulfide (FeS), which can interfere with vivianite formation. When the S/Fe ratio is higher than 1.1, vivianite formation is greatly impeded (Rothe *et al*., 2015) [27].
4. **Microbial activity is also significant:** Through reduction of ferric ions to ferrous and release of phosphate from organic matter, dissimilatory iron-reducing bacteria (DIRB) like Shewanella oneidensis and Geobacter sulfur reducens facilitate vivianite synthesis (Newsome *et al*., 2018; Wang *et al*., 2020) [20, 36]. Sulfate-reducing bacteria (SRB), however, may hinder the process by forming sulfide (Rothe *et al.,* 2016) [26]. The microbial enzyme-dependent conversion of organic phosphorus into soluble orthophosphate (Monbet *et al*., 2007) [19].
5. **The saturation index (SI):** A supersaturation measure, controls the rate at which crystals form and grow. While a SI greater than 11 causes spontaneous (homogeneous) nucleation, a SI greater than 4 enhances precipitation. Vivianite develops optimally at pH levels near 7, where SI reaches a metastable condition.
6. **Organic compounds:** i.e., sodium alginate (SA) and humic acid (HA), could inhibit vivianite generation. The shape and size of the crystals are modified by this interaction, which also reduces phosphorus recovery (Zhang *et al*., 2020) [48]. Increased initial pH and Fe/P ratios, however, could reduce this negative effect.
7. **Temperature:** With increase in temperature, vivianite formation get reduced.
8. **Alkalinity:** With addition of alkali to waste water increases the formation of vivianite.

Lastly, by lowering the supersaturation level, seed materials facilitate the initiation and growth of vivianite crystallization. Besides facilitating the growth of larger crystals, application of such materials as sponge iron facilitates magnetic separation, which enhances the efficiency of the reactor and reduces chemical consumption (Yu *et al*., 2013; Wu *et al*., 2021) [44, 30].
Finally, hydraulic conditions and current density also affect vivianite formation. Optimal upflow velocity ranges from 30.56 to 68.76 m/h in fluidized bed systems (Priambodo *et*

al., 2017a) [23], while electrochemical P removal benefits from current densities above 7.5 mA/cm² (Bektaş *et al*., 2004) [2].

Vivianite applications in Agriculture

1. Slow-Release Phosphorus Fertilizer: Vivianite is a slow-release phosphorus source, giving prolonged nutrient supply to crops. Because it has low water solubility, vivianite does not leach easily into the environment, thereby minimizing nutrient loss and environmental degradation. The primary advantages are phosphorus release control over an extended period of time. Frequency of fertilization reduced. Lower eutrophication risk because of limited P runoff

The fertilizer effect of two recycled P products obtained from water purification: vivianites and struvites. The struvites performed similarly to superphosphate and outperformed all types of vivianites in terms of dry matter (DM) yield and P uptake. Industrial and synthetic vivianites led to higher dry matter (DM) yield and P uptake by plants than vivianites from water purification. Synthetic and industrial process vivianites increased Olsen P in soils after harvesting. Vivianite from industrial process can replace 54-75% of superphosphate on a DM basis (Ayeyemi *et al*., 2023) [1]. The highest phosphorus recovery was found at a Fe/P ratio of 1.8. Below the 24-h settling time, the phosphorus removal efficiency was significantly lower. The phosphorus removal efficiency of vivianite crystallization obtained in the CM-C reactor was maximum of 69% and FB-C reactor had P recover efficiency of 67% (Nurdan *et al*., 2023) [21]. The microbially mediated vivianite as a novel phosphorus (P) fertilizer for wheat where total P uptake by wheat plants from the product dominated by vivianite and phosphate-green rust (VivSol) was not significantly different from potassium dihydrogen phosphate (KH_2PO_4). The relative P use efficiency of VivSol was 74% of KH_2PO_4, making VivSol the effective P source for durum wheat (Eshun *et al*., 2024) [8].

2. Soil Amendment and Iron Supplement

Not only is vivianite a phosphorus provider but also a source of iron (Fe^{2+}), which is a critical micronutrient for plant metabolisms. Its dual-nutrient function has the ability to correct iron deficiencies in calcareous or alkaline soils (Wilfert *et al*., 2016) [40].

3. Application in Organic and Sustainable Agriculture

Since vivianite is recoverable from wastewater and sewage sludge, it follows circular economy and sustainable agriculture principles. It provides a sustainable substitute for mined rock phosphate, which decreases reliance on non-renewable sources of P (Zhang *et al.,* 2022) [47].

4. Co-application with Organic Matter

Research indicates that vivianite can be co-applied with compost or other organic manures to increase soil's capacity for nutrient retention and microbial populations, resulting in better soil fertility and plant productivity (Zhou *et al*., 2018) [49].

Constraints and limitations in vivianite utilization

Most existing technologies are still in the pilot or laboratory scale, with high cost of operations and intricate arrangements, particularly for such processes as electrochemical crystallization and anaerobic digestion (Zhang *et al*., 2022) [47]. Last but not least, vivianite has commercial competition from struvite (magnesium ammonium phosphate) that is more regularly recovered and known in wastewater treatment. Even with its greater value and wider potential for applications, vivianite remains less researched and utilized, which restricts its usage in the present to real-world applications (Zhang *et al.,* 2022) [47].

Future prospectives

Vivianite production from wastewater is a highly promising method for sustainable phosphorus recovery and recycling of resources. Future research will likely be directed towards enhancing recovery efficiency using innovative technologies such as electrochemical systems, microbial treatment, and magnetic separation. Growing demand for vivianite use in fertilizers, battery materials (e.g., $LiFePO_4$), and environmental restoration increases its commercial value. Also, real-time monitoring technologies and friendly policies encouraging circular economy application will spur developments. Still, sulfide interference issues, costs of the processes, and system optimization need to be solved to allow wide-scale applications.

Conclusion

In conclusion, since rock phosphate is in short supply and food requirements continue to increase, vivianite recovery from sewage sludge and wastewater presents a workable and profitable solution to the global phosphorus deficit. Vivianite offers a circular economy solution to nutrient stewardship through its numerous applications in farming, such as a soil conditioner, iron supplement and slow-release fertilizer. Recovery efficiencies have been significantly enhanced by advances in recovery methods, such as chemical precipitation, electrochemical systems, biotreatments, adsorption and also by an improved understanding of key influencing parameters, like pH, Fe/P ratios, and microbial activity. Process scalability, cost, sulfide interference and limited commercialization remain concerns, however. Vivianite's complete potential as a next-generation phosphorus recovery product will be reliant on continued research, favorable regulations, and the integration of advanced technologies.

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