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# Research Progress on the Characteristics and Recovery of Phosphorus from Sludge

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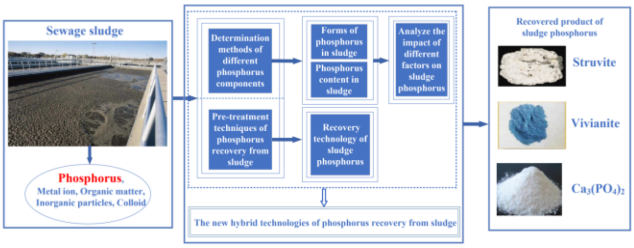
## Graphical abstract




## Abstract

Phosphorus is a non-metallic element essential for all forms of life, and the issue of phosphorus resource scarcity is increasingly gaining attention. Understanding the content and forms of phosphorus in sludge, as well as the technologies for phosphorus recovery from sludge, is of great importance. Firstly, this paper provided a comprehensive overview of various pre-treatment methods (biological, oxidation, acid-base, microwave, ultrasonic, and hydrolysis) for sludge phosphorus recovery, highlighting the potential for combining these techniques to enhance recovery efficiency. Secondly, it introduced the concept of mixed recovery technology for phosphorus in sludge, which involved the strategic arrangement and combination of different treatment technologies. This approach represented a novel and innovative way of addressing the phosphorus recovery challenge. Furthermore, by discussing the effects of various factors on the phosphorus content in sludge, the paper offered an integrated perspective on the complex interplay of these factors, facilitating a more nuanced understanding of the phosphorus recovery process. This review provides a good reference for recovery processes of sludge phosphorus, enabling engineers to select the most appropriate techniques based on specific project requirements.

**Keywords:** Phosphorus Characteristics, Sludge, Phosphorus Release, Phosphorus Recovery

## 1. Introduction

Phosphorus is an essential nutrient for all living organisms, particularly for crop growth. However, the global scarcity of phosphorus resources presents a significant concern (Cooper *et al.* 2011), estimated to sustain human usage only until the end of this century. Therefore, it is of utmost necessity to conserve phosphorus resources and find viable pathways for their recovery. In human production and daily life, a substantial amount of phosphorus enters wastewater treatment plants along with sewage, and approximately 1/6 of the phosphorus from phosphate rock mining also ends up in wastewater treatment facilities via industrial wastewater (Cordell *et al.* 2009). Consequently, the recovery of phosphorus from sewage sludge has become an important method for phosphorus resource conservation. In particular, waste activated sludge can contain up to 10% of its dry weight in phosphorus, making it a significant source of this nutrient (Tchobanoglus *et al.* 2003). The efficient recovery of phosphorus from sludge presents a current challenge. This paper summarized and compared the characteristics and recovery methods of phosphorus in sludge and discussed future development directions.

## 2. The Influencing Factors and Forms of Phosphorus in Sludge

### *2.1. Content and Forms of Phosphorus in Sludge*

The total phosphorus content in sludge is approximately 17.3±5.1g/kg (Wang *et al.* 2018). Phosphorus in sludge exists in two forms: inorganic phosphorus and organic phosphorus. In dehydrated sludge, inorganic phosphorus accounts for 60% to over 90% of the total phosphorus content, primarily existing in mineral forms such as pyrophosphate, polyphosphate, orthophosphate, and calcium/aluminum-phosphate. Inorganic phosphorus is further divided into apatite and non-apatite forms, with the former mainly combining in the form of Ca-P and the latter primarily combining with Fe/Al/Mn oxides or hydroxides (Wang *et al.* 2023). Organic phosphorus accounts for 10% to 35% of the total phosphorus content in sludge, mainly existing in the form of orthophosphate monoesters, orthophosphate diesters, adenosine triphosphate, and adenosine diphosphate α-phosphates. In wastewater treatment plants, the proportion of inorganic phosphorus gradually increases in sludge produced from primary settling tanks, secondary settling tanks, and digesters

(Kwapinski *et al.* 2021). The forms of phosphorus in sludge are directly related to the phosphorus removal methods employed by municipal wastewater treatment plants. In 2001, the European Commission proposed a unified procedure for phosphorus extraction, defining five identifiable forms of phosphorus: total phosphorus (TP), organic phosphorus (OP), inorganic phosphorus (IP), non-apatite inorganic phosphorus (NAIP), and apatite phosphorus (AP). The five forms of phosphorus in sludge have the following relationships: IP = NAIP + AP, TP = IP + OP. IP is the predominant form of phosphorus, accounting for 64.42% to 83.01% of TP; NAIP is the main form of inorganic phosphorus, accounting for over 63% of IP (Zhu *et al.* 2014).

### *2.2. The Influence of Different Factors on the Forms of Phosphorus in Sludge*

The forms of phosphorus in sludge vary with different treatment methods; both wastewater treatment (including phosphorus removal methods and the composition of the original wastewater) and sludge treatment influence the forms of phosphorus.

During the wastewater treatment process, the phosphorus contained in the wastewater impacts the outcome; additionally, different wastewater treatment technologies result in various forms of phosphorus in the subsequent sludge, such as the combination of metal ions in the wastewater with phosphates (Zhang and Kuba, 2014).

Sludge treatment technologies lead to various forms of phosphorus, causing a decrease in OP and polyphosphates (poly-P), and an increase in IP (Liu *et al.* 2019). Acid treatment reduces the content of non-apatite phosphorus (e.g., iron and aluminum phosphates) and apatite phosphorus (calcium phosphate), whereas alkaline treatment decreases phosphate content and increases apatite phosphorus levels. Additionally, thermal treatment processes yield various effects. Hydrothermal (HT) treatment not only reduces OP and poly-P and increases IP but also alters the distribution of IP subtypes, namely, Fe\P and Ca\P distributions. The higher the temperature in HT treatment, the greater the production of Ca\P (Biswas *et al.* 2020). After incineration, the phosphorus in sludge ash primarily exists as $Fe_4(P_4O_{12})_3$ and $Al(PO_3)_3$ (Biswas *et al.* 2009); varying incineration temperatures significantly influence the transformation of phosphorus forms (Xiao *et al.* 2021). Sludge incineration typically occurs at about 850°C, where phosphorus exists as volatile oxides in the flue gas and condenses into $P_4O_{10}$ compounds upon cooling to 400~600°C (Meng *et al.* 2019). Factors influencing the migration and transformation of phosphorus during the pyrolysis process include sludge characteristics and heating rate, among others (Zhang, 2020). Specifically, as sludge particle size decreases, AP content increases, and when the particle size exceeds 37.5μm, NAIP content tends to decrease (Li *et al.* 2017). Pyrophosphates are found in slow pyrolysis biochar produced at temperatures between 300~700°C, whereas orthophosphates are absent in the rapid carbonization products of sludge at 350°C (Uchimiya *et al.* 2015).

### *2.3. Measurement Methods for Different Phosphorus Components in Sludge*

The primary research approach for detecting phosphorus in sludge involves converting phosphorus into water-soluble phosphorus compounds (Zeng *et al.* 2023), followed by measuring its content. For example, Zhou Xuhong *et al.* (Zhou and Cao, 2005) melted sludge samples with sodium hydroxide at high temperatures to convert all phosphorus-containing organic compounds into soluble orthophosphates; Chen Guomei *et al.* (Chen, 2006) used potassium persulfate digestion to convert phosphorus into orthophosphates; Kuai Lijun *et al.* (Kuai *et al.* 2019) employed microwave digestion to transform TP in sludge into orthophosphates. In all cases, the TP content determination was conducted using the ammonium molybdate spectrophotometric method.

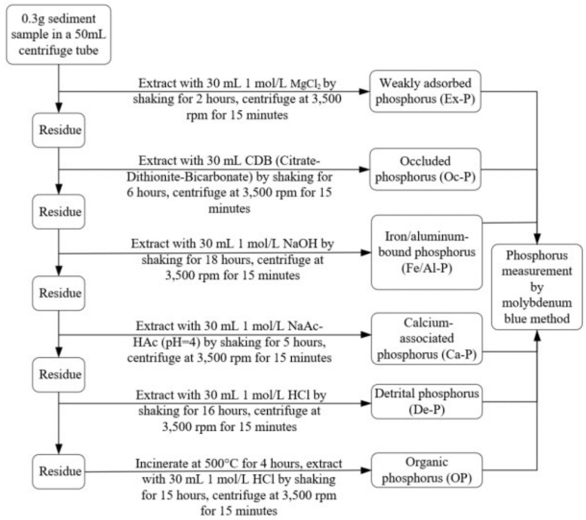



**Figure 1.** Sequential Extraction of Phosphorus

The measurement methods for different types of phosphorus in sludge generally involve phosphorus fractionation extraction followed by determination using the molybdenum blue method, as illustrated in **Figure 1**. This method can be economical, rapid, and accurate identify the types of phosphorus. However, the qualitative analysis of phosphorus species may lack clarity, affecting the accuracy of subsequent quantitative analysis (Yu *et al.* 2021). The determination of phosphorus components can also be conducted using specific instrumental analysis methods, such as X-ray diffraction (XRD), $^{31}P$ nuclear magnetic resonance spectroscopy, and X-ray absorption near edge structure (XANES) spectroscopy. Compared to colorimetric methods, these instrumental analyses can provide more detailed information on species and components. However, the use of inappropriate pretreatment methods may result in the loss of key phosphorus species (Yu *et al.* 2021). Currently, an improved $^{31}P$ nuclear magnetic resonance method can prevent the hydrolysis of polyphosphates during characterization (Staal *et al.* 2019), and an enhanced $PL_{2,3}$-edge XANES method can identify the subtypes of IP and OP more accurately (Colocho *et al.* 2020).

## 3. Current Status of Phosphorus Recovery Technologies from Sludge

### *3.1. Pre-treatment Technologies for Phosphorus Recovery from Sludge*

Pre-treatment technologies for phosphorus recovery involve using biological, oxidation, acid-base, microwave, ultrasonic, and hydrolysis methods to facilitate the breakdown of extracellular polymeric substances and cell lysis in sludge. This process leads to the release of organic matter, nitrogen, phosphorus, and other substances, making them more accessible for subsequent phosphorus recovery. Currently, the primary pre-treatment technologies for phosphorus recovery from sludge were summarized in **Table 1**.

**Table 1.** Pre-treatment Technologies for Phosphorus Recovery from Sludge

| Main Technologies | Overview | Affecting Factors and Treatment Outcomes | Current Limitations |
|---|---|---|---|
| Biological Method - Anaerobic Digestion | Under anaerobic conditions, microorganisms decompose solid matter, releasing intracellular substances and phosphorus (Sun *et al.* 2014), with the phosphorus being primarily in the form of inorganic phosphates (Wang *et al.* 2020). | Anaerobic digestion is optimal with a 5-day sludge age; but metal ions hinder the process (Dong *et al.* 2020). | Long response time, high infrastructure costs. |
| Oxidation Method | The oxidation method uses strong oxidants to break down microorganisms and release organic matter, which is further oxidized to release phosphorus. Ozone is the most widely used oxidant. | Below 61.2 mg $O_3$/gSS, total phosphorus and orthophosphate concentrations in ozonated sludge rise with increasing ozone dosage (Zhang *et al.* 2017). | An excessively high ozone dosage can result in the oxidation of dissolved components, thereby reducing the apparent solubility. |
| Acid-Base Method | The acid-base method involves the addition of strong acids or strong alkali reagents to disrupt cell osmotic pressure and extracellular polymers, leading to cell lysis and facilitating the conversion of particulate organic matter in sludge into dissolved organic matter (Yuan *et al.* 2010). | Phosphorus release is pH-dependent, with lower solubilization under acidic than alkaline conditions (Chen *et al.* 2007). | The acid-base method requires excessive acid or alkali, complicating phosphorus separation and recovery. |
| Hydrothermal Method | Hydrothermal treatment, by heating sludge, breaks down flocs and cell walls, rapidly releasing phosphorus (Ahmad and Idris, 2013), and is a key pretreatment method for anaerobic digestion. It also improves the biodegradability of sludge, methane yield, and dewatering performance (Wang *et al.* 2016). | The optimal temperature for the hydrothermal method ranges from 50~150°C, with the optimal treatment time being 40~60 minutes (Zhu *et al.* 2014). | High-temperature hydrolysis consumes much energy, risks scaling in the reactor, and can yield toxic compounds that hinder phosphorus recovery and utilization (Xu *et al.* 2017). |
| Microwave Method | The microwave method utilizes thermal heating and non-thermal effects to break down sludge. | Increasing temperature, altering biomolecules, and changing physicochemical properties enhance total phosphorus release, improve sludge dewatering, which is more effective on primary sludge than on mixed or digested sludge. (Wojciechowska, 2005). Microwave irradiation, at 200~600W, achieves enhanced phosphorus release effects with a relatively short treatment time (Fang *et al.* 2021). | Microwave irradiation is energy-intensive and poses potential health risks (Fu *et al.* 2019), which limits its practical application in sludge pre-treatment. |

| | | | |
|---|---|---|---|
| Ultrasonic Method | The ultrasonic method employs the cavitation effect of ultrasonics as a mechanical technique to break down sludge flocs, cell bodies, and aggregates (Wang *et al.* 2018), releasing substances such as phosphorus into the liquid phase. | The optimal ultrasonic amplitude ranges from 20~10MHz (Wang *et al.* 2018), with phosphorus release varying significantly among different sludge types under the same conditions (Wang *et al.* 2010). | Due to its high energy consumption and the need for complex equipment, the ultrasonic method is not economically viable in real-world industrial settings. |

### *3.2. Phosphorus Recovery Technologies from Sludge*

Phosphorus recovery technologies from sludge involve methods to recover phosphorus released into the supernatant after pre-treatment, utilizing adsorption, ion exchange, membrane filtration, and chemical or struvite precipitation techniques. The primary technologies for phosphorus recovery from sludge were summarized in **Table 2**.

**Table 2.** Main Technologies for Phosphorus Recovery from Sludge

| Main Technologies | Overview | Examples | Current Limitations |
|---|---|---|---|
| Adsorption Method | The adsorption method uses adsorbents to capture substances like nitrogen and phosphorus from solution, followed by desorption for separation, involving chemical and physical adsorption, as well as deposition on solids. | The magnetic adsorbent Zr improved can effectively adsorb and release phosphorus (Lin *et al.* 2020). | The adsorption method frequently encounters challenges like adsorbent poisoning, limited regeneration performance, and the high costs of adsorbents (Xiao *et al.* 2022). |
| Ion Exchange Method | The ion exchange method utilizes the exchange groups of ion exchange resins to swap with phosphate ions in the solution, thereby accomplishing phosphorus recovery (Mei *et al.* 2013). | Weak base resins preferentially remove phosphates over other anions and support repeated, long-term use for phosphate extraction from water (Awual and Jyo, 2011). | The ion exchange method is limited in its target range and is currently best suited for low-concentration phosphorus solutions (Mei *et al.* 2013). |
| Membrane Filtration Method | Nanofiltration retains heavy metals and recovers phosphorus using screening and electrostatic effects (Ding *et al.* 2022). Nanofiltration membranes work by selectively excluding multivalent metal cations while capturing neutral and negatively charged phosphate compounds in the permeate (Schütte *et al.* 2015). | With a pH of 0.5 and a solution permeability of 90%, phosphorus recovery can reach 83.7% (Schütte *et al.* 2015). | Most current research is at the laboratory stage, requiring further exploration of practical application and cost-effective membrane material development (Ding *et al.* 2022). |
| Chemical Method | Hydrolysis reaction of metal salt compounds produces hydroxyl complexes, effectively reducing or eliminating the colloidal ζ potential (Xing *et al.* 2022), and result in the formation of phosphate precipitates. | OriorGutierrez (Gutierrez *et al.* 2010) found that the amount of precipitated phosphorus was related to the added iron salt. | The chemical method can effectively lower the phosphorus content in high-concentration wastewater, but recovering phosphorus from the precipitates is challenging. |
| Magnesium Ammonium Phosphate Precipitation Method | Magnesium ammonium phosphate ($MgNH_4PO_4 \cdot 6H_2O$), also known as struvite or MAP, recovers phosphorus by adding magnesium salts to sludge supernatant rich in ammonium ions and phosphates (Liu *et al.* 2023). | The optimal pH range for the struvite crystallization reaction is 8~9 (Liu, 2009); the best recovery effect is achieved with a $Mg^{2+}:NH^{4+}:PO_4^{3-}$ ratio of 1.15:1:1 (Zhang *et al.* 2009). | Phosphate compounds of iron, calcium, and aluminum, with high solubility products, tend to precipitate first, resulting in higher struvite recovery costs and lower phosphorus recovery rates (below 50%) under complex conditions (Wilfert *et al.* 2015). |

### *3.3. New Technologies for Phosphorus Recovery from Sludge*

#### *3.3.1. Recovery of Sludge Phosphorus in the Form of Vivianite*

Vivianite, a ferrous (Fe(II)) phosphate mineral, was discovered during the anaerobic digestion (AD) of wastewater sludge (Wilfert *et al.* 2016). In a reducing environment rich in organic matter, Fe(III) is reduced to

Fe(II) by dissimilatory iron-reducing bacteria, converting organic phosphorus into phosphates (Rothe *et al.* 2016). Subsequently, the free Fe(II) combines with phosphates to form vivianite, as illustrated in **Figure 2**. Vivianite is widely utilized as the final phosphorus product in phosphorus recovery from iron-rich sludge (Hu *et al.* 2023) and can serve as a slow-release fertilizer. Its efficiency is significantly superior to that of calcium phosphate, positioning it as an ideal alternative to traditional fertilizers. Additionally, vivianite serves as a fundamental source for manufacturing lithium iron phosphate ($LiFePO_4$) and is used in lithium battery production (Zhao *et al.* 2023).

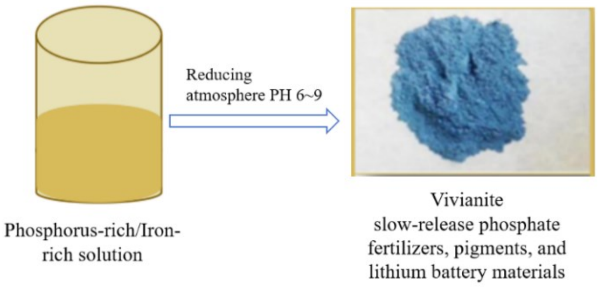



**Figure 2.** Formation of Vivianite in Iron and Phosphorus-Rich Solutions

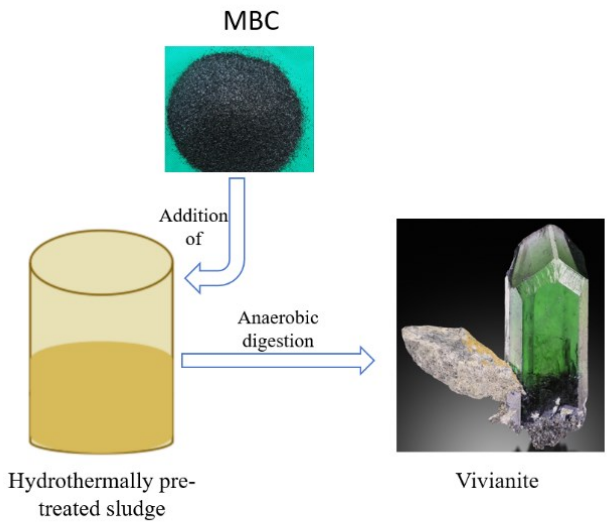



**Figure 3.** Magnetic Biochar Promoting the Anaerobic Digestion Process of Sludge Pre-treated Hydrothermally

At present, vivianite formation in the AD of sludge is hindered by the low molar ratio of Fe/P in sludge. Magnetic biochar (MBC) is added as an external iron source during the AD process, altering the iron/phosphorus concentration in the sludge. MBC enhances the anaerobic digestion process of hydrothermally pre-treated sludge, offering a novel technology for recovering sludge phosphorus in the form of vivianite, as depicted in Figure 3. The most favorable iron/phosphorus molar ratio for vivianite formation ranges from 1.5 to 3. Furthermore, MBC, with its large surface area and rich porosity, offers enhanced adsorption capacity for phosphates and additional growth sites for microorganisms (like Fe(III) reducing bacteria), facilitating the combination of phosphates with Fe(II) and thus promoting vivianite formation (Liu et al. 2022). HT can hydrolyze cellulose to glucose, and subsequent glucose hydrolysis generates reactive furfural derivatives, which can reduce Fe(III) to Fe(II) (Jiang et al. 2015). The most suitable conditions for forming the maximum amount of vivianite include HT pre-treatment at 135°C, the addition of MBC, and ten days of anaerobic digestion (Liu et al. 2022).

### *3.3.2. Anaerobic Fermentation Combined with Sulfate Reduction (AF-SR) System for Phosphorus Recovery*

The AF-SR system enables the simultaneous release of polyphosphates and iron-phosphates from wasted activated sludge. It has been revealed that the total phosphorus release rate primarily depends on the generation of sulfides (Hu *et al.* 2019). The environmental conditions of the AF process are conducive to the growth of sulfate-reducing bacteria (SRB) (Weng *et al.* 2015). Concurrently, the volatile fatty acids produced by AF can serve as a carbon source for SRB, and the sulfides produced by SRB can be consumed by iron, preventing the accumulation of $H_2S$ and its inhibitory effects on SRB (Hu *et al.* 2019).

Combining anaerobic digestion of wasted activated sludge with in-situ sulfate reduction offers an alternative method for recovering phosphates from precipitates containing iron phosphate. This method can utilize waste streams containing sulfates, such as bagasse or wastewater from paper mills, as auxiliary raw materials (Rodrigues and Hu, 2017). Phosphorus recovery can be achieved through two strategies (Likosova *et al.* 2013): the first involves directly treating iron-containing pre-coagulated wasted activated sludge or synthetic iron phosphate suspensions with NaHS or $Na_2S$, which releases phosphates from solid precipitates into the liquid phase. The second method promotes the sulfate reduction process under anaerobic conditions through sulfate-reducing bacteria, thereby releasing phosphates from wasted activated sludge into the liquid phase. This method not only increases the concentration of phosphates in the liquid phase but also maintains the anaerobic digestion process in a suppressed and stable state. Although this approach may lead to a decrease in methane production, it ensures that the output remains at a stable level. Phosphate recovery is economically feasible, but it requires integration with energy, organic matter, or other resources for the comprehensive utilization of energy-rich sludge (Lippens and De Vrieze, 2019).

### *3.3.3. Alkali Combined with Microwave Pre-treatment of Sludge and Cross-linked Polymer for Phosphorus Recovery*

Combining alkali and microwave pre-treatment of sludge, followed by adding methyl triethoxysilane (MTAC) to recover organic matter and nitrogen-phosphorus in the supernatant, represents a novel approach to sludge phosphorus recovery. MTAC can cross-link with water to create polyorganosiloxanes, which exhibit good biocompatibility (Grabitz *et al.* 2020). The silanol groups, produced through the hydrolysis of alkoxy groups, can attach to inorganic materials via covalent bonds (Wang *et* *al.* 2018), making them effective for phosphorus recovery. Alkali treatment, a widely used chemical pre-treatment method, is often combined with other processes. The thermal and non-thermal effects generated by microwaves can efficiently break down sludge. Therefore, the combination of alkali and microwave pre-treatment offers significant potential. The release of phosphorus through alkali treatment combined with microwave radiation increased by up to 25.13% compared to alkali treatment alone (Dai *et al.* 2023). Ultimately, combining alkali and microwave pre-treatment with cross-linked polymer technology recovers substances such as phosphorus from the sludge supernatant in the form of flocculated aggregates. The resulting flocculated aggregates, rich in organic components and nitrogen-phosphorus nutrients, with abundant surface pores and stability, show potential as slow-release carbon sources, fertilizers, and soil conditioners (Dai *et al.* 2023).

### *3.3.4. Acidic Phosphorus Recovery (APR) Process*

Ruo-hong Li *et al.* (Li *et al.* 2019) developed a novel biological treatment process named the APR process, which effectively combines trivalent Fe(III)-based chemical phosphorus removal technology with Membrane Bioreactor (MBR) technology. This approach aims to enhance wastewater treatment efficiency and recover phosphorus from sludge effectively. This process uses an environmentally friendly approach to recover phosphorus from iron-containing sludge produced by the Chemical Phosphorus Removal (CPR) and CPR-MBR treatment processes (Lin *et al.* 2017). Compared to the traditional CPR method, the APR process extracts and recovers phosphorus from iron-containing sludge by co-fermenting it with kitchen waste to produce acid. This synergistic fermentation process takes place under acidic conditions, where trivalent Fe(III) is reduced to divalent Fe(II) through dissimilatory reduction. This leads to the dissolution of iron-phosphorus complexes in sludge and the release of phosphorus (Li *et al.* 2019). This process effectively transforms waste into valuable resources, offering a sustainable solution for sludge phosphorus recovery.

### *3.3.5. $FeCl_3$-Assisted Hydrothermal Carbonization (HTC) Process for Sludge Treatment*

The $FeCl_3$-assisted HTC process for sludge treatment selectively leaches AP and recovers phosphorus as $KMgPO_4 \cdot 6H_2O$. During the HTC process, organic phosphorus, polyphosphates, and pyrophosphates are converted into orthophosphates (Li *et al.* 2019). The abundant presence of apatite and other calcium salts in sludge hinders effective phosphorus recovery. Adding ferric chloride creates an appropriate acidic condition for dissolving AP and also immobilizes the dissolved phosphorus to form NAIP (Li *et al.* 2020). Utilizing the $FeCl_3$-assisted HTC process transfers and fixes orthophosphates into hydrochar, facilitating sewage sludge reduction. Phosphorus on the hydrochar is then extracted into the aqueous phase and recovered as K-struvite (Li *et al.* 2020). This technology standardizes phosphorus extraction conditions from hydrochar, achieving a total phosphorus extraction efficiency of 96.1%. Introducing amphoteric hydroxide masking agents and external magnesium recovers over 92% of the system's total phosphorus as K-struvite. Additionally, this treatment method hydrolyzes the organic matter in the sewage sludge, resulting in a sludge volume reduction of approximately 43-55%.

## 4. Conclusion

Given the global depletion of phosphorus resources, the phosphorus shortage has emerged as a critical issue. Sludge, a significant reservoir of phosphorus, is continuing to attract attention. Nonetheless, existing technologies for phosphorus recovery from sludge are challenged by high costs, unstable recovery efficiency, and the risk of secondary pollution. Consequently, further improvements and innovations are required as follows:

(1) A deeper understanding of the intrinsic mechanisms and the organic integration of various technologies for a complementary and multidimensional approach could lead to more efficient and stable phosphorus recovery.

(2) The focus should extend beyond the technology for recovering phosphorus from sludge to optimizing the wastewater treatment systems of sewage treatment plants. By leveraging modern technologies, integrating the entire sewage sludge treatment with intelligent control systems, and utilizing big data models, real-time monitoring and optimization of the entire treatment process can be realized. This approach would enhance the overall efficiency and economic benefits of phosphorus recovery and facilitate the comprehensive utilization of resources.

(3) With the expanding market for sludge-derived phosphorus, technological updates and supportive policies are necessary. Strengthening the cooperation between governments and enterprises to jointly address the global phosphorus resource issue is crucial.

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