

Cognizing and characterizing the organic phosphorus in lake sediments: Advances and challenges

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ABSTRACT

Organic phosphorus (OP) is one of the main forms of phosphorus in lake ecosystems. Mounting evidence has shown that sediment OP has become a major but underestimated issue in addressing lake eutrophication and algal bloom. However, a holistic view of sediment OP remains missing. This review aims to provide an overview of progress on the studies of OP in lake sediments, focusing on the contribution of OP to internal P loading, its potential role in algal bloom, and the migration and transformation. In addition, this work systematically summarized current methods for characterizing OP content, chemical fraction, composition, bioavailability, and assessment of OP release in sediment, with the pros and cons of each method being discussed. In the end, this work pointed out following efforts needed to deepen the understanding of sediment OP, namely: (1) In-depth literature review from a global perspective regarding the contribution of sediment OP to internal P loading with further summary about its pattern of distribution, accumulation and historical changes; (2) better mathematical models for describing drivers and the linkages between the biological pump of algal bloom and the replenishment of sediment OP; (3) fully accounting the composition and molecular size of OP for better understanding its transformation process and mechanism; (4) developing direct, high-sensitivity and combined techniques to improve the precision for identifying OP in sediments; (5) establishing the response of OP molecular properties and chemical reactivity to OP biodegradability and designing a comprehensive and accurate composite index to deepen the understanding for the bioavailability of OP; and (6) integrating fundamental processes of OP in current models to better describe the release and exchange of P in sediment-water interface (SWI). This work is expected to provide critical information about OP properties and deliver perspectives of novel characterization methods.

1. Introduction

Lake eutrophication presents a significant global challenge for human health and ecological protection (Brookes and Carey, 2011; McCrackin et al., 2017). Phosphorus (P) has been regarded as a limiting factor for eutrophication and is responsible for the large-scale burst of cyanobacteria growth (Carpenter, 2008; Conley et al., 2009). Sediment is the major P pool in lake systems, which can significantly affect water quality by P releasing (Nürnberg et al., 2013; Markovic et al., 2019). Efficient P reduction from sediments has become a major research topic in lake management as strict external P discharge limits have been

implemented. The release and diffusion flux of P calculated by thermodynamics and kinetics model of soluble reactive P (SRP) are the basis for assessing the risk of sediment P release (Tohru et al., 1989; Fan et al., 2002). By comparison, organic P (OP) has a similar magnitude as inorganic P (IP) in many aquatic ecosystems (Björkman and Karl, 2003; Ding et al., 2010; Feng et al., 2018), but it is usually ignored due to the complexity regarding its composition, bioavailability, environmental processes, and effects (Bai et al., 2009; Shinohara et al., 2017). In recent years, there is an increasing emphasis on the importance of OP to lake eutrophication, water quality and algal blooms, with numerous studies on OP composition, bioavailability, fate, behavior and environmental impacts (Dyhrman et al., 2006; Bünenmann, 2008; Duhamel et al., 2021;

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Nomenclature

OP	Organic phosphorus
SWI	Sediment/water interface
SRP	Soluble reactive P
DOP	Dissolved organic phosphorus
DOC	Dissolved organic carbon
DON	Dissolved organic nitrogen
POP	Particulate organic phosphorus
IP	Inorganic P
DO	Dissolved oxygen
APA	Alkaline phosphatase
DOM	Dissolved organic matter
MW	Molecular weight
LMW-DOM	Low molecular weight dissolved organic matter
HMW-DOM	High molecular weight dissolved organic matter

ICP-MS	Inductively coupled plasma mass spectrometry
³¹ P NMR	³¹ P nuclear magnetic resonance
FT-IR	Fourier transform infrared
XANES	X-ray absorption near-edge structure
FT-ICR-MS	Fourier transform ion cyclotron resonance mass spectrometry
EDTA	Ethylenediaminetetraacetate
DBE	Double bond equivalent
Bio-OP	Bioavailable organic phosphorus
Res-OP	Residual organic phosphorus
α -gly	α -glycerophosphate
β -gly	β -glycerophosphate
Glu-6	D-glucose 6-phosphate
IHP6	Inositol hexakisphosphate
RNA-mnP	RNA mononucleotides
Pyro-P	Pyrophosphate

(Ni et al., 2021). Since 1980s, 181 articles on sediment OP have been published on the Web of Science. The number is about a quarter of published works on soil OP and about 8.3% of works on P, with approximately ten new articles per year since 2010 (Fig. 1).

OP contains a variety of different compounds ranging from nucleotide, phospholipids, phosphates, phosphor-sugar, phosphoprotein, inositol phosphate, to organic complex phosphate (Worsfold et al., 2008; Venkatesan et al., 2018). The other components, with no distinct chemical formulae or cannot be dissolved by chemicals, are usually referred to as refractory OP. OP represents an important bridge between the biogeochemical cycles of P and biological processes (Duhamel et al., 2021). For example, phytoplankton can directly use low molecular weight (LMW) OP for reproduction (Zou et al., 2005), while plants (e.g., clover, barley, oats and wheat) are able to utilize OP efficiently as they do with inorganic sources (Tarañdar and Claassen, 1988). For nutritional utilization, dissolved organic phosphorus (DOP) is much more labile than dissolved organic carbon (DOC) and nitrogen (DON) in the marine environments (Lønborg and Álvarez-Salgado, 2012), with the mineralization rate of DOP being nearly twice as rapidly as DOC and DON in the ocean (Letscher and Moore, 2015). In lake sediments, OP is usually closely related to lake trophic level and is considered a better indicator than total P for trophic level and sediment pollution level (Vaalgamaa, 2004; Liao et al., 2010; Ni et al., 2019a). In particular, the bioavailable OP is found to regulate eutrophic status and facilitate algal bloom of

lakes (Zhu et al., 2013; Ni et al., 2019b; Darch et al., 2013). Those studies imply that OP might have an ecological and environmental impact on aquatic systems. Therefore, lake management and protection plans must consider the role, contribution, and potential risks of OP in water quality and eco-environment.

In recent years, the development of analytical techniques for OP structure, composition and bioavailability has received a great attention to better understand the degradability and the release of OP in sediments. Nonetheless, no systematic studies have been performed to investigate the characteristics of OP, and a substantial portion of OP components remains unresolved (McLaren et al., 2015; 2020). Furthermore, the biogeochemical cycle and key pathways of OP, such as adsorption-desorption, ligand exchange, mineralization, humification, photolysis, enzymatic hydrolysis and molecular diffusion, have not received comprehensive measures. As a result, in-depth discussions about the importance and the potential role of sediment OP in lake eutrophication and algal bloom are lacking, with inconclusive or even controversial findings about the content, chemical fraction, composition, bioavailability, migration and transformation of OP.

Several review literature have summarized studies which look into the determination of aquatic OP (Worsfold et al., 2008; Darch et al., 2013), chemical nature of soil OP (McLaren et al., 2020), soil OP mineralization rates (Bünemann, 2015; Martínez, 1968), as well as the distribution, circulation of OP and its ecological significance to the aquatic environments (Baldwin, 2013). However, studies on the potential role of sediment OP in lake eutrophication, algal blooms, the migration and transformation pathways remain inadequate, with few works focusing on improving the characterization methods of OP using multiple methods despite that they might provide critical insights for fathoming the biogeochemical behavior of sediment OP.

This review aims to fill those gaps by: (1) elaborating the contribution of OP to sediment internal P loading and its potential role in algal blooms, (2) updating the latest progress on the studies about migration and transformation pathways and mechanisms of sediment OP, and (3) summarizing and critically discussing the state-of-art characterization methods for sediment OP. Built on those basis, we further put forward the challenges, issues and opportunities that might shed light on further research. This work is expected to broaden our understanding on the characteristics and environmental impact of OP in lake sediments, and deliver new perspectives about the characterization methods of OP.

2. Contribution of OP to sediment internal P loading

As a major component in lake P biogeochemistry, OP exists widely in water body, macrophytes, phytoplankton, particulate matter, atmospheric deposits, and sediments (Fig. 2). In rivers, it was found that DOP

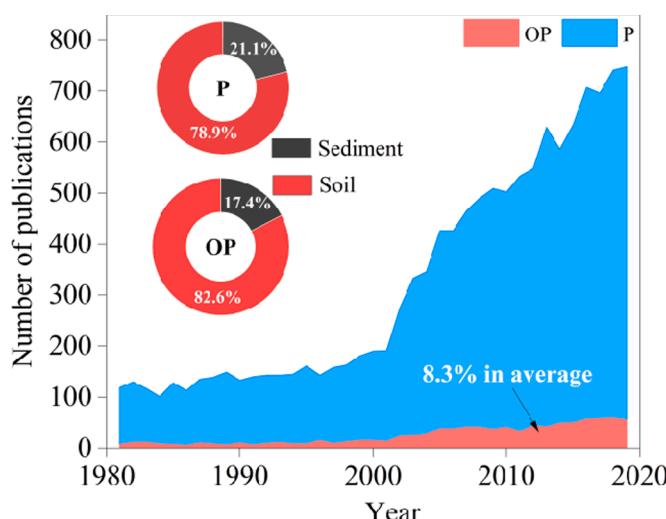


Fig. 1. The number of published articles on "P" and "OP" in sediments and soil since 1980s. (Source: Web of Science).

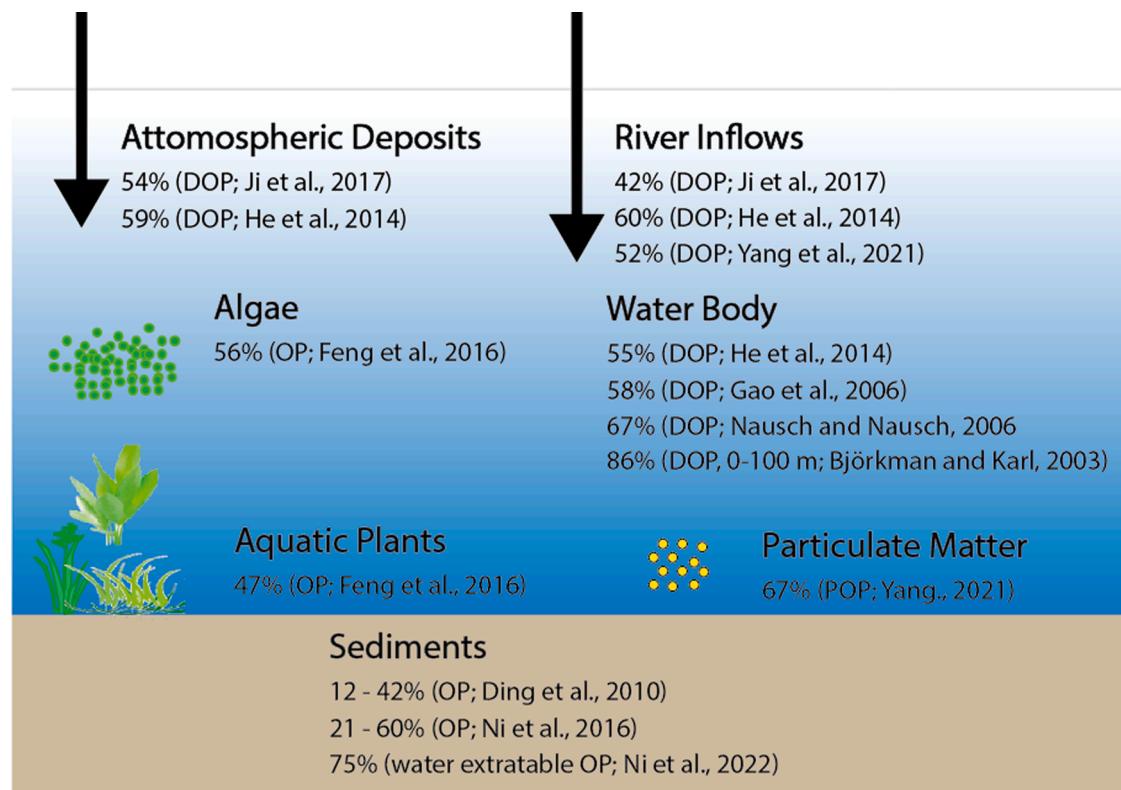


Fig. 2. Typical proportions of OP on total P in river inflow, water bodies, macrophytes, phytoplankton, particulate matter, atmospheric deposits, and sediments from aquatic ecosystems, respectively.

contributed up to 38–68% (42% on average), 4.2–100% (60% on average), and 39–69% (52% on average) of the total dissolved P (DTP) in Erhai Lake (Ji et al., 2017), Dianchi Lake (He et al., 2014) and the Great Lakes (Yang et al., 2021), respectively. In water bodies, the concentration of DOP accounts for more than 55% of DTP in Taihu Lake, Dianchi Lake and Baltic Sea (Nausch and Nausch, 2006; Gao et al., 2006; He et al., 2014), and it was also reported that the DOP pool was often 5–8 times larger than the measured SRP pool in water bodies (0–100 m below the surface) in the North Pacific Subtropical Gyre (Björkman and Karl, 2003). The particulate OP (POP) in Fox River and the Great Bay of the Great Lakes accounts for 47–87% (67% on average) of total particulate P (Yang et al., 2021). The DOP in atmospheric deposits on average accounts on average for 55% and 59% of DTP in Erhai Lake and Dianchi Lake of China, respectively (Ji et al., 2017; He et al., 2014). For aquatic plants, OP could account on average for 56% and 47% of total P in algae and aquatic macrophytes, respectively (Feng et al., 2016). Two surveys have showed that, for Chinese lakes, the content of OP to total P can vary from 12 to 60% (Ding et al., 2010; Ni et al., 2016). In addition, the water extractable OP was found to be the dominant part in DTP (74.5% on average) of the sediments from ten Chinese lakes (Ni et al., 2022a). These results indicate the high proportion of OP in aquatic systems, thus the contribution of sediment OP to internal P loading cannot be ignored.

It is worth to notice that, during past decades the drastic recessions of aquatic vegetations (Zhang et al., 2017) and increased phytoplankton bloom events (Ho et al., 2019) has led to the vast amount of aquatic vegetations and phytoplankton debris, which accelerate the accumulation of OP in lake sediments worldwide. Wastewater from residential areas and agricultural activities (e.g., domestic sewage treatment, application of chemical fertilizers and manure, livestock farming) is another factor that contribute to OP loading on lakes (Lü et al., 2016; Ni et al., 2020). The accelerated P loss from agricultural activities and residential wastewater in the past decades (Liu et al., 2016; Yuan et al., 2018), especial in developing countries, would enhance OP

accumulation in lake sediments, and thus cause water quality issue and aquatic biodiversity losses.

Studies have shown that the enhanced lake eutrophication level will significantly increase the content of enzymatic hydrolysable OP in sediments (Ni et al., 2016), while hypereutrophic lakes with high algal biomass will accelerate OP hydrolysis and mineralization (Yuan et al., 2020), which indicates that, with the continuously intensified lake eutrophication, the accumulated OP in sediments might become a ticking time bomb that will jeopardize lake water quality in future. Wang et al. (2008) reported that polluted sediments correspondent to high release of DOP from sediments. Furthermore, the contribution from OP mineralization in sediments to eutrophication is found to be greater than that of IP in macrophyte-dominated eutrophic lakes, and the mineralization process plays a critical role in the release of internal P (Yu et al., 2022). These results reflect that, with global accelerated lake eutrophication and enhanced anthropogenic P inputs, it is urgent to investigate the accumulation and the contribution of sediment OP to internal loading to prevent the deterioration of lake ecosystems.

3. Potential role of sediment OP in algal blooms and eutrophication

Algae act as a primary link between P circulation and lake ecosystems (Zou et al., 2020). It is known that algal blooms could enhance the release of Fe-bound P from sediments, which in turn provide SRP to support algal blooms (Xie et al., 2003; Chen et al., 2018; Wang et al., 2019a; Yao et al., 2022), forming a positive feedback loop between sediment P flux and algal blooms, so-called biological pump.

For OP, some research reported that algae (e.g. *prorocentrum donghaiense* Lu) and bacterial (e.g., *M. aeruginosa*) can use DOP compounds efficiently via alkaline phosphatase (APA) to sustain its growth under SRP depletion (Huang et al., 2005; Harke et al., 2012). That is, DOP utilization is also vital in shaping phytoplankton community in P-deficient waters (Ma et al., 2019). APA plays an important role in regulating

algal blooms and P dynamics in aquatic environments (Wang et al., 2021). The activity of APA in sediments is usually influenced by the outbreak and perishing of algae, with APA being more active during algal blooms (Xu et al., 2013; Bao et al., 2015; Zhang et al., 2015). The enhanced transformation of SRP from DOP by elevated cell-associated APA activity and quantity in eutrophic water and sediment would replenish bioavailable P to further promote algal blooms (Yuan et al., 2019; Wang et al., 2021), and the capacity of OP hydrolysis is close to that of SRP uptake under P-starvation (Moorleghem et al., 2013). Furthermore, the remineralization of DOP can increase the niche for diazotrophs and facilitate nitrogen fixation (Letscher and Moore, 2015), thus providing more nitrogen source for algae.

Lake eutrophication processes and algal bloom can also significantly alter aquatic conditions, such as pH and dissolved oxygen (DO). Zuo (2015) reported that the release of OP from sediments significantly increased under pH from 7 to 9.5, and DO level appeared to be the most influential in terms of OP release from sediments, with sediments under anoxic conditions generally releasing more OP than that of aerobic environment (Zuo, 2015; Zhang et al., 2017). This suggests that high pH and low DO level during algal blooms would promote the release of OP from sediments. The settled algae residues could enhance the accumulation of Fe-P and bioavailable OP in sediments, which in turn replenish available P pools to further facilitate algal growth (Ni et al., 2019b; Wang et al., 2019a). Like sediment IP, this positive feedback between the accumulation and release of sediment OP and algal growth would sustain algal blooms persistently even under controlled external P loading or low level of SRP in water. However, the quantitative relationship between algal blooms and the replenishment of OP in sediments remains unclear. Therefore, better mathematical models or methods for describing the linkage between the biological pump of algal bloom processes (i.e. cyanobacteria formation, aggregation and decay processes) and the replenishment (i.e. dynamic process of degradation, transformation and diffusion) of OP in sediments would be an impending issue (Fig. 3).

4. Migration and transformation of OP in sediments

The migration and transformation of OP in sediments mainly include adsorption, desorption, deposition, diffusion, biological assimilation, mineralization, humification and the transformation of chemical composition and molecular size (Fig. 4). Like adsorption/desorption mechanism of orthophosphate, OP can absorb with minerals (e.g., iron aluminum oxide) through ligand exchange (Olsson et al., 2010; Yan et al., 2014a, 2014b). Some species of OP (e.g., denosine monophosphate, adenosine triphosphate, glucose-6-phosphate and

aminoethylphosphonic acid) showed lower efficiency in sorption with iron oxide than that of orthophosphate (Ruttenberg and Sulak, 2011), indicating that the mobility of these DOP compounds may exceed that of orthophosphate in sediments under certain conditions. In contrast, the adsorption quantity of iron aluminum oxide and clay mineral with inositol hexaphosphate are much larger than that of orthophosphate due to the abundant phosphate groups of inositol hexaphosphate (Yan et al., 2012), which might explain its high stability in sediments. The migration of OP from sediments to overlying water involves four pathways: (1) chemical process, namely the degradation of OP releases through chemical hydrolysis (Li et al., 2011) and photolysis (Li et al., 2019), (2) biological process, namely biomineralization (Wang and Pant, 2010), (3) the kinetic process, namely the resuspension of POP and DOP under the action of wind, hydrodynamic force, benthic activities (Li et al., 2019), and (4) the diffusion of DOP between water-sediment interface (Guan et al., 2013).

The dynamic change of chemical composition is the most important indicator for OP transformation. Previous studies suggested that the compositional transformation of OP in sediments is usually accompanied by the degradation, release, and burial of P compounds. For example, Yu et al. (2022) found that the mineralization of sediment OP can result into P-combination Fe-Al/Fe-Mn. Phospholipid is usually degraded into α -glycerophosphate and β -glycerophosphate (Doolette et al., 2009), while RNA-P can turn into nucleotide (Bünemann et al., 2008) by microorganisms under alkaline conditions. Also, some OP species such as phytate (myo-inositol-1,2,3,4,5,6-hexakisphosphate) could be transformed into orthophosphate by phytase hydrolysis (Zhu et al., 2013) or by precipitation as metal complexes form into insoluble and stable metal-phytate, thus buried in sediments permanently (Jørgensen et al., 2011). Therefore, elucidating the transformation of OP chemical composition would provide critical information for understanding the biogeochemical cycle of OP and for assessing its potential risk from sediments.

Another under-explored area is about the molecular size transformation of OP. Recent research revealed that the compositional transformation of organic molecules is usually accompanied by changes of molecular size, while the intrinsic transformation and degradation of organic molecules is usually regulated by both molecular size and organic compositions (Xu and Guo, 2018). For instance, the concentrations of total organic carbon, carbohydrates, and protein-like substances decrease during dissolved organic matter (DOM) incubation, while the MW of DOM would transform from LMW-DOM ($< 1 \text{ kDa}$) to high MW-DOM (HMW-DOM)/colloids ($1 \text{ kDa} - 0.45 \mu\text{m}$) and further transform into microparticles ($> 0.45 \mu\text{m}$) (Xu and Guo, 2018). Liberated or freshly photosynthesized DOM and DOP, generated by assimilation and uptake of SRP via phytoplankton, would be mainly composed by HMW substances or colloids (Qu et al., 2012; Benner and Amon, 2015; Yang et al., 2021), while the HMW organic molecules are usually have higher biological reactivity and degradation rates than its LMW counterparts (Benner and Amon, 2015; Ni et al., 2022a). This might be explained by the fact that HMW-DOM/colloids can either be aggregated into particles by colloidal pumping and self-assembly (Honeyman and Santschi, 1989; Xu and Guo, 2018), or be decomposed into smaller molecules through photolysis, enzymatic hydrolysis, or chemical reactions (Santschi et al., 2006; Santschi, 2018). The transformation of molecular size exerts a profound impact on adsorption/desorption as well as the bioavailability of OP, which impose the potential risk of P release from sediments. For instance, the maximum sorption of DOP with iron (oxyhydr)oxides decreases with the increasing MW of P compounds (Ruttenberg and Sulak, 2011; Yan et al., 2014a). Shinohara et al. (2017) argued that the large molecular size of molybdenum unreactive P in pore water hampers its diffusion from sediments into overlying water. Therefore, the composition and the dynamic changes of molecular size of OP shall be taken into account to better explore the transformation mechanism, and the potential environmental impact of OP in sediments.

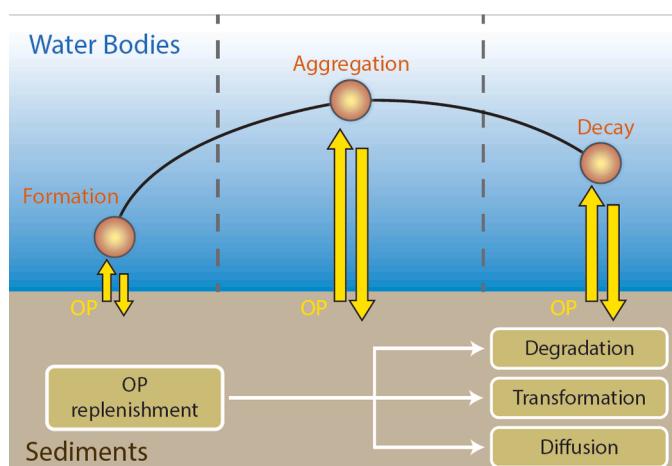


Fig. 3. A schematic relationship between the biological pump of algal bloom processes and the replenishment of OP in sediments.

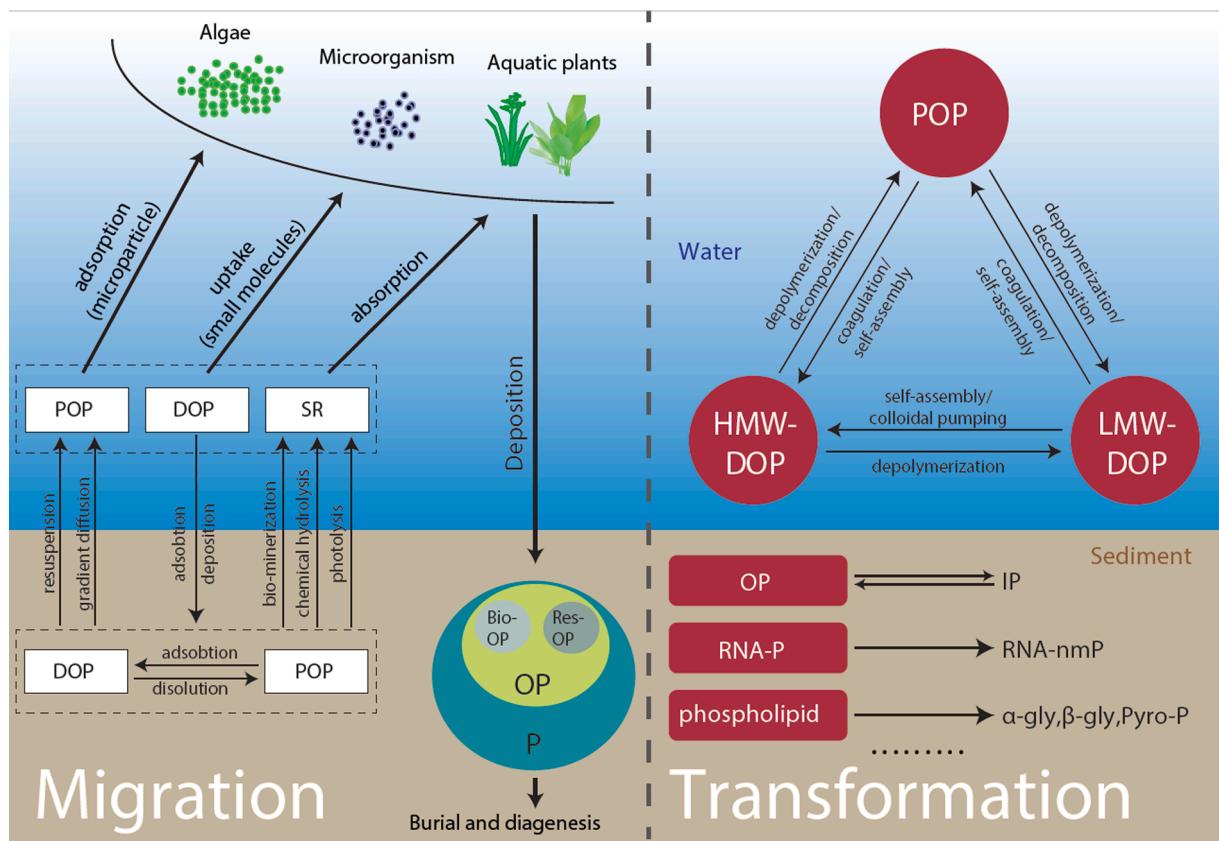


Fig. 4. Major paths of migration (left side) and transformation (right side) of OP in sediments.

5. Advances in characterization methods of sediment OP

Characterizing sediment OP is a key to understand its properties and to determine its environmental behavior, fate, as well as potential environmental risk. This section reviewed the state-of-the-art characterization methods and technologies for content, chemical fraction, composition, structure, bioavailability and release in sediment OP.

5.1. OP contents

Typically, the content of OP in sediments is determined by chemical extraction and burning methods (Anderson, 1960). The content is calculated as the difference between DTP and SRP after extracted by chemical extracts. However, SRP usually includes inorganic P and acid hydrolysable organics (Duhamel et al., 2021), which would underestimate the content of OP if extraction method is used. In comparison, during the digestion procedure the pyrophosphate, metaphosphate and polyphosphate in DTP would transform into orthophosphate, leading to the over-estimation of the OP. For example, Turner et al. (2006) found that molybdate colorimetry significantly overestimated the content of OP by between 30 and 54% relative to NMR spectroscopy results due to the influence of association of inorganic phosphate with organic matter.

Furthermore, some OP species might be chemically hydrolyzed with strong acidic or alkaline solutions during extraction process (Degroot and Golterman, 1993; Worsfold et al., 2008; Turner et al., 2003), or be volatilized during thermal hydrolysis and burning processes (Huang et al., 2017), especially for compounds with high OP content (Lu, 2000). Therefore, developing direct and high-sensitivity methods are essential to better quantify the contents of OP in sediments.

5.2. OP chemical fractionation

Fractionation is a useful approach for separating complex organic

compounds into homogeneous fractions (He and Hur, 2015), such that one can analyze the geochemical reactivity of each fraction and their roles in environment. Traditional sequential extraction scheme presumes that chemical extractants can dissolve specific species of OP, and it remains a major approach to estimate the fractions of OP compounds in sediments. Table 1 summarized the development of extraction schemes for OP in soils and sediments. Sommers et al. (1972) first raised the method for identifying HCl-OP, cold NaOH-OP and hot NaOH-OP in OP. Later, a systematical fractionation procedure was proposed by Bowman and Cole (1978) to further identify labile, moderately labile, moderately resistant, and highly resistant OP fractions, respectively. Built upon these basis various other fractionation methods have been put forward (Hedley et al., 1982; Tissen and Moir, 1993; Ivanoff et al., 1998; Golterman, 1996; Golterman et al., 1998; Zhu et al., 2013).

The common extractants in OP fractionation include H_2O , $NaHCO_3$, HCl and NaOH. In general, H_2O and $NaHCO_3$ can be used to extract microbially labile organic molecules (Cross and Schlesinger, 1995; Guigue et al., 2016; Zhang et al., 2008; Textor et al., 2019), while alkali solution can extract humics and fulvic acids combined P (Ivanoff et al., 1998; Chen and Hur, 2015). During acidification fulvic acids remain soluble, but humics will precipitate when the pH is below 2 (Ivanoff et al., 1998; McDonald et al., 2004). Sequential OP fractionation presumes that H_2O -OP and $NaHCO_3$ -OP fractions can represent labile OP, while HCl-OP and NaOH-OP (fulvic acid-OP) are usually correspondent to moderately labile OP (Ivanoff et al., 1998). However, Zhu et al. (2013) and Ni et al. (2019b) demonstrated that in lake sediments the bioavailability (enzymatic hydrolysis rate) of H_2O -OP and $NaHCO_3$ -OP were less than that of HCl-OP and NaOH-OP, which suggested that the assessment of OP activity with sequential fractionation may be inaccurate. Furthermore, some studies have shown that strong NaOH is not an accurate extractant as its concentration and duration influence the quantity of OP via chemical hydrolysis (Golterman et al., 1998), while the tedious and complex extraction process will also introduce

Table 1

Major chemical extraction schemes for OP fraction in soils and sediments.

No.	Author	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Developed for
1	Sommers et al., 1972	1 N HCl, 25 °C HCl-OP	0.3 N NaOH, 25 °C Cold NaOHOP	0.3 N NaOH, 90 °C Hot NaOHOP	/	/	/	/	Sediment
2	Bowman and Cole, 1978	0.5 M NaHCO ₃ , pH 8.5 Labile OP	1 M H ₂ SO ₄ Moderately labile OP	0.5 M NaOH, pH 1~1.8, Not precipitated Moderately resistant OP	0.5 M NaOH, pH 1~1.8, precipitated Highly resistant OP	/	/	/	Soil
3	Hedley et al., 1982	Deionized water, resin, Resin-P	0.5 M NaHCO ₃ NaHCO₃-OP	CHCl ₃ Microbial-OP	0.1 M NaOH NaOH-OP	1 M HCl HCl-OP	/	/	Soil
4	Tiessen and Moir, 1993	Deionized water, resin, Resin-P	0.5 M Na HCO ₃ NaHCO₃-OP	0.1 M NaOH NaOH-OP	Concentrated HCl HCl-OP	/	/	/	Soil
5	Ivanoff et al., 1998	0.5 M NaHCO ₃ , pH 8.5 NaHCO₃-OP	1 M HCl + 0.5 M NaOH, pH 2 Not precipitated Fulvic acid-OP	1 M HCl + 0.5 M NaOH, pH 2 Precipitated Humicacid-OP	Ashing, 1 M H ₂ SO ₄ Residual OP	/	/	/	Soil
6	Golterman, 1996; Golterman et al., 1998	Water H₂OOP	0.05 M Ca-EDTA, pH 7.5 Fe-OP	0.1 M Na-EDTA Ca/Al-OP	0.5 M H ₂ SO ₄ Aid soluble-OP	0.5 M TCA 0 °C Nucleic acid-OP	0.5 M TCA 95 °C Sugar-OP	2 M NaOH 90 °C Humic-OP	Sediment
7	Zhu et al., 2013	Water H₂OOP	0.5 M NaHCO ₃ pH 8.5 NaHCO₃-OP	0.1 M NaOH NaOH—OP	1 M HCl HCl-OP	/	/	/	Sediment

large errors in results. Last but not least, the mutual interference and poor specificity among different extractants make it difficult to assign the environmental significance to P fractions (Turner and Leytem, 2004; Liu et al., 2013). Therefore, the results derived from traditional sequential fractionation schemes should be interpreted with cautions, and it is urgent to implement novel fractionation methods based on modern characterization techniques.

5.3. OP composition and structure

The characterization of OP composition and structure is critical in studying sediment OP. Thanks to the recent development of modern spectroscopy, mass spectrometry and chromatography, there have been encouraging results in characterizing the OP's composition and structure at molecular levels (Fig. 5). These analytical techniques include inductively coupled plasma mass spectrometry (ICP-MS) (Shah and Caruso, 2005), ³¹P nuclear magnetic resonance (³¹P NMR) spectroscopy (Carman et al., 2000), Fourier transform infrared (FT-IR) spectroscopy

(Yang et al., 2015), soft X-ray fluorescence spectromicroscopy (Brandes et al., 2007), X-ray absorption near-edge structure (XANES) spectroscopy (Hurtarte et al., 2019), and Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR-MS) (Brooker et al., 2018).

Solution ³¹P NMR spectroscopy is the most frequently used one because its non-destructive feature (Cade-Menun, 2005), which allows to identify monoester P (e.g., α/β-glycerophosphate, nucleotide, D-glucose 6-phosphate, phytate and, choline phosphate), diester P (e.g., DNA-P, RNA-P, and phospholipids), and phosphonate, etc. However, the ethylenediaminetetraacetate (EDTA)-NaOH extraction and the enrichment process in the ³¹P NMR pretreatment would cause chemical degradation of OP compounds, leading to the underestimation of OP content in sediments (Ni et al., 2022b). FT-IR spectroscopy is another non-destructive method, which provides real-time information regarding the vibration of P chemical bond and the functional groups of DOMs (Yang et al., 2015). For instance, the principal peaks of the FT-IR spectra at 1033–1030, 876–873 and 797–779 usually associate with phospholipids, DNA and RNA-P (P=O bond), aromatic (P-O-C bond),

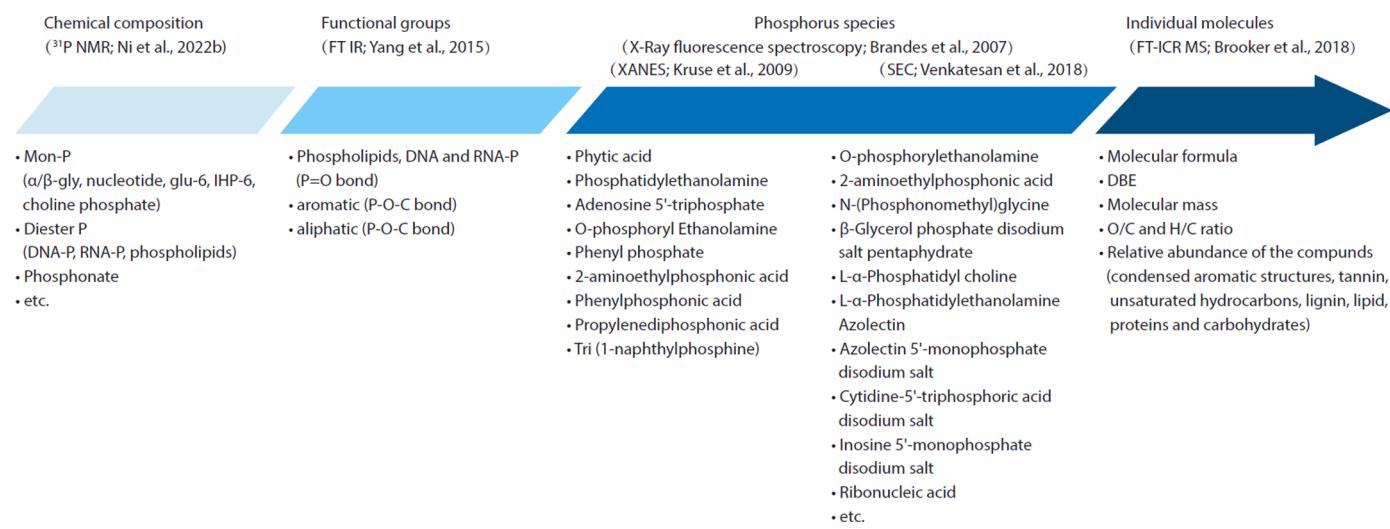


Fig. 5. Development of common characterization methods for the composition and structure of OP compounds.

and aliphatic (P-O-C bond), respectively (Ni et al., 2019a). However, if samples contain complex compounds with a similar structure, their spectroscopy peaks will be overlapped and the compounds will be indistinguishable (He et al., 2006).

X-ray fluorescence spectroscopy and microscopy are used for element abundance analysis and speciation characterization with up to parts-per-trillion level sensitivities (Twining et al., 2003, 2004), which holds the promise for analyzing sediment OP. Brandes et al. (2007) examined a range of P containing organic phases in marine sediments using X-ray fluorescence spectroscopy. The identified OP species includes phytic acid, phosphatidylethanolamine, adenosine 5'-triphosphate, O-phosphorylethanolamine, phenyl phosphate, 2-aminoethylphosphonic acid, phenylphosphonic acid, propylenediphosphonic acid, Tri(1-naphthylphosphine).

XANES spectroscopy allows direct identification of organic and mineral P species, with no pre-extraction and minimal sample pre-treatment (Hesterberg, 2010; Kizewski et al., 2011). The OP compounds featuring energy location of P L_{2,3}-edge XANES spectra mainly include O-phosphorylethanolamine, 2-aminoethylphosphonic acid, N-(phosphonomethyl) glycine, β-Glycerol phosphate disodium salt pentahydrate, 1-α-Phosphatidyl choline, 1-α-Phosphatidylethanolamine, asolectin, adenosine 5'-monophosphate disodium salt, cytidine-5'-triphosphoric acid disodium salt, inosine 5'-monophosphate disodium salt, ribonucleic acid, phytic acid sodium salt hydrate, and phytic acid calcium salt (Kruse et al., 2009). A limitation of XANES spectroscopy is that many OP compounds' spectra are indistinguishable (Eriksson et al., 2015).

FT-ICR-MS is a new and robust method for characterizing the composition and structure of DOM, which allows the detection of various DOM components up to thousand species based on their molecular formulas (Ohno et al., 2014). The identified parameters include double bond equivalent (DBE), molecular formula, molecular mass, O/C and H/C ratio, as well as the relative abundance of OP compounds (e.g., condensed aromatic structures, tannin, unsaturated hydrocarbons, lignin, lipid, proteins and carbohydrates). However, due to the low element abundance of P in DOM and the interference of inorganic salts, there are only a few studies to date that attempt to apply FT-ICR MS method to investigate the molecular composition and structural characteristics of DOP in environment (Brooker et al., 2018; Ni et al., 2021; Gao et al., 2021; Kurek et al., 2020).

The limitation of each method indicates that, one technique alone cannot adequately characterize OP. Instead, combined analytical techniques might offer a promising way. Liu et al. (2013) adopted sequential fractionation, solution ³¹P NMR, and P K-edge XANES spectroscopy together to investigate soil OP species and provided comprehensive information about P species. Kurek et al. (2020) analyzed sediment OP constitutes using different chemical extractants in combination with FT-ICR MS, and suggested that milder extractants resulted in better OP formulae assignments than stronger extractants. Venkatesan et al. (2018) employed size exclusion chromatography coupled with ICP-MS to characterize the MW distribution of OP in water. He found that the size exclusion chromatography in combination with ICP-MS and DOC offers a powerful and easy way for identifying many unknown P fractions. Liu et al. (2015) is the first one to determine the variations of legacy P species in soils using sequential fractionation, ³¹P NMR and XANES spectroscopic techniques. McLaren et al. (2015) and Ni et al. (2022b) used solution ³¹P NMR spectroscopy and MW ultrafiltration to reveal the dynamics of the MW distribution of OP components, which provided more detailed information about OP composition and bio-reactivity in soil/sediments. Wang et al. (2019b) proposed a method that combined liquid chromatography in tandem with mass spectrometry to quantitatively determine six most important phosphonates species in effluent of wastewater treatment plant. Thus, future studies should focus on establishing multiple combined analytical systems to characterize sediment OP from various perspectives.

5.4. OP bioavailability

The bioavailability of OP is an important indicator for assessing the potential contribution of OP to lake eutrophication. Bioavailability refers to the relative ease of a specific OP to be biologically degraded and assimilated into microbial biomass (Duhamel et al., 2021). It is closely related to the microbial capacity of uptake, assimilation and sorption, as well as the APA activities (Berg and Joern, 2006). Popular methods for OP bioavailability include bio-culture (i.e. bacteria, algae, and hydrophyte) and enzymatic hydrolysis. In practice, some researchers found that the environmental conditions of bacteria and algae culture are difficult to control, and the results contain a large uncertainty due to biological differences and the high heterogeneity of time and space (Duhamel et al., 2021). In addition, most of the culture experiment chose a specific biological species as a tester, which cannot reflect actual environmental conditions.

Enzymatic hydrolysis has been widely used to estimate the potential bioavailability of OP, since most OP must be hydrolyzed into SRP before it is utilized by organisms (Bünemann, 2008). Although this method is easy to apply, it suffers from poor precision and reproducibility due to the complexity in environmental conditions as well as in species and the quantity of enzymes. Moreover, the enzymatic hydrolysis method may not reflect actual biodegradation in natural environment because: (1) APA can be strongly adsorbed by minerals (e.g., montmorillonite and goethite) in sediments under natural conditions (Zhu et al., 2016), and thus decrease the activity of APA; (2) APA activities may be inhibited by humic acids, fluoride, metal ions and polyvalent anions in natural environment (Wang and Pant, 2010; Freitas et al., 2014); and (3) the commonly used enzymes are derived from terrestrial organisms, e.g., bovine intestinal mucosa (alkaline phosphatase), crotalus atrox (phosphodiesterase), and wheat (phytase), which cannot reflect actual enzymatic activities in real aquatic environment. Instead, using more diversified aquatic enzymes will be more representative and thus can better assess aquatic OP bioavailability (Duhamel et al., 2021).

Molecular properties are the primary control on the microbial utilization of dissolved organic molecules in aquatic systems (Shen and Benner, 2020; 2022). An understanding of the relationship between OP molecular characteristics and its availability will be beneficial for evaluating the response of natural communities to different OP compounds (Fig. 6). Molecular characteristics usually involve composition, structure, hydrophobicity/hydrophilicity, MW, humification degree, the number of functional groups, and stoichiometric ratio, etc. For example, labile OP species (e.g., diester P, phospholipids, and teichoic acids) are mineralized within a short period of approximately 3 days while other OP (e.g., monoester P and phosphonates) with in 4 and 13 weeks (Darch et al., 2014). Qin et al. (2015) found that the hydrophobic DOP fraction with a low C/P ratio was more bioavailable to algal growth in effluent DOP. Gao et al. (2021) reported that DOP species with low oxygen content, low aromaticity and low unsaturation were preferentially degraded by biological treatments in wastewater treatment plant. Ohno et al. (2014) found that the DOM components with H/C > 1.2 and O/C > 0.5 are strongly correlated with biodegradability in forest soil. Yang et al. (2022) reported that within the less-available DOP compounds in runoff and surface waters, lignin-like compounds are dominant. Ni et al. (2022a) found that the bioavailable DOP were closely related to MW, with the contents of DOP from enzymatic hydrolysis decreased with decreasing molecular weight, which is consistent with the findings by Amon and Benner (1996) and Benner and Amon (2015), who reported that HMW-DOM are more bio-reactive. Crecchio and Stotzky (1998) and Zhu et al. (2015) reported that humics combined OP is usually resistant to enzymatic hydrolysis, indicating that the higher degree of humification DOM in sediments, the less enzymatic hydrolysis of DOP it will be. Letscher and Moore (2015) found that DOP is remineralized about twice as rapidly with respect to enriched C:N stoichiometry of marine DOM.

Therefore, to further understand the bioavailability of OP it is necessary to establish the response of molecular properties and the

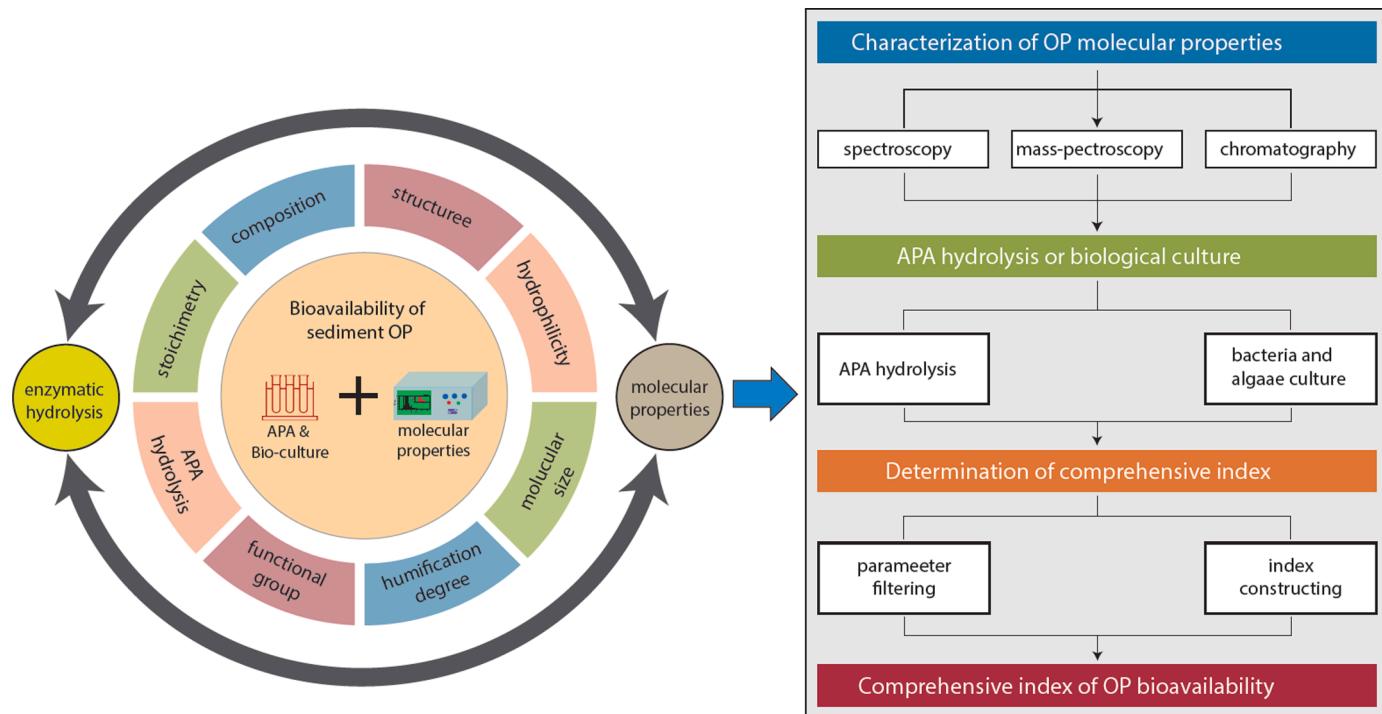


Fig. 6. Proposed research steps for establishing a comprehensive and accurate composite index to deepen the understanding for the bioavailability of sediments OP.

chemical reactivity to biodegradation of OP, and the construction of a more comprehensive and accurate composite index that account key molecular characteristics and biodegradation parameters to estimate the bioavailability of OP would be a promising topic to explore.

5.5. Quantitative assessment of OP release in sediments

Reducing the release of P from sediments to water is one of the major approaches in ecological restoration of lakes due to its low cost and simplicity (Horppila, 2019), while how to assess the performance is a key. At present, different methods have been developed focusing on assessing the release and exchange of P in sediment-water interface (SWI). Those methods include mass balance models (Kelderman et al., 2005), sediment incubation (Du et al., 2018; Wang et al., 2018a), benthic chamber experiment (Buchholz-ten Brink et al., 1989), molecular diffusion models (Lavery et al., 2001), reaction and transformation models (Doan et al., 2018), and adsorption-desorption kinetics models (Wang et al., 2018b), etc. Although OP is abundant in sediments, current methods for P release and diffusion in SWI only focus on SRP, where OP is usually ignored (Mandal et al., 2015; Shinohara et al., 2017). The difficulty in modeling sediment OP release can be attributed to its complex composition, bioavailability, and transformation processes.

Shinohara et al. (2017) constructed a model of OP release from sediments according to the mathematical relationships among the degradation and mineralization of OP, as well as the adsorption, desorption, and diffusion of IP in SWI. They pointed out that the model needs to further consider the concentration and flux of preferentially degradable OP components (e.g., diester P and some mono-P species), key environmental factors affecting enzyme activity and P release, and the effect of molecular size on the release rate of P.

Given the importance of OP to sediment P loading, OP need to be integrated within current models of P release and exchange in SWI. Possible elements include: (1) the mineralization and degradation coefficient of OP with different components, MW and hydrophobicity, (2) the diffusion coefficient of DOP in pore water with different MW, and (3) the estimation of bioavailability of OP that from direct gradient

diffusion, as well as (4) the key environmental conditions (e.g., pH value, DO level, temperature, sunlight irradiation, APA activity, microorganisms). A holistic consideration of these elements will deepen our understanding of the methods and approaches for OP release, and is conducive to further improve the accuracy in assessing OP release in sediments.

6. Conclusion and outlook

This review provides an overview of current studies with respect to OP in lake sediments, with a focus on the contribution of OP to internal P loading, its potential role in algal blooms, the migration and transformation, as well as its characterization methods. OP has a predominant role in the migration and transformation of P in lake ecosystems, while the enhanced accumulation of sediment OP and its increased contribution to the internal P loading appears to be a major concern under global accelerated lake eutrophication and anthropogenic P inputs. The positive feedbacks among the degradation and release of OP to algal blooms might act as important reasons to explain the persistence of algal bloom events. Investigating the migration and transformation paths of OP in SWI using combined methodologies and multiple perspectives is a key to studying the biogeochemical cycle of OP.

In addition, this work systematically summarized current characterization methods for OP content, chemical fraction, composition, bioavailability, and quantitative assessment of OP release in sediments, with the pros and cons of each method being discussed. To sum up, following efforts are needed to deepen the understanding of OP in lake sediments (Fig. 7):

- (1) In-depth literature review from a global perspective regarding the contribution of sediment OP to internal P loading with further summary about its pattern of content, distribution, accumulation, historical changes. In addition, future studies should provide predictions about the accumulation and potential risk of OP release from sediments under the background of the global and regional trends of lake eutrophication, anthropogenic activities, and climate.

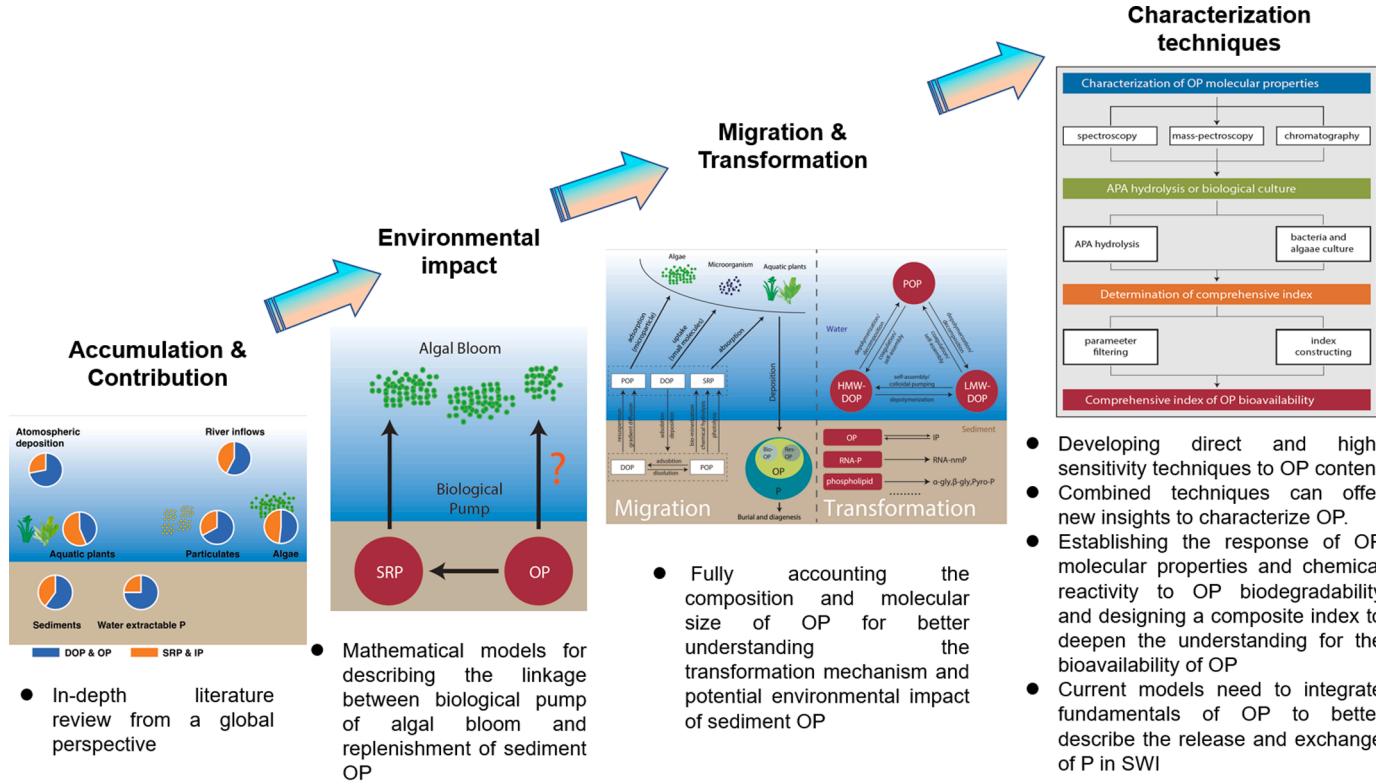


Fig. 7. Research outlook of potential topics for OP in lake sediments.

- (2) Better mathematical models for describing the linkage between the biological pump of algal blooms and the replenishment of OP in sediments. One should explore the drivers of the positive feedback loop among algal blooms, the migration transformation and partition of OP in SWI. The coupling effect of multiple factors on the replenishment of sediment OP during algal bloom period should also be better illustrated.
- (3) Fully accounting the composition and molecular size of OP is conducive for better understanding its transformation process and mechanism.
- (4) Developing direct and high-sensitivity techniques to improve the precision for identifying OP content in sediments. The assessment of OP chemical activity by traditional sequential fractionation may be inaccurate, and it is urgent to implement novel methods for characterizing sediments OP fractions based on modern characterization techniques. A single technique cannot provide comprehensive assessment of OP composition and structure due to the limit of each method, while combined techniques can offer new insights.
- (5) Deepening the understanding for the bioavailability of OP requires establishing the response of OP molecular properties and chemical reactivity to OP biodegradability, while a comprehensive and accurate composite index would be a promising approach.
- (6) Current models need to integrate fundamentals of OP to better describe the release and exchange of P in SWI. Those elements include the mineralization and degradation coefficient of different OP, the diffusion coefficient of DOP, the bioavailability of OP from gradient diffusion, and key environmental conditions.

Declaration of Competing Interest

The authors declared that they have no conflicts of interest to this work. We declare that we do not have any commercial or associative interest that represents a conflict of interest in connection with the work

submitted in this article.

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