



Analyses of phosphorus in sewage by fraction method

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ABSTRACT

Several forms of the phosphorus in wastewater were analyzed using a fraction method with a membrane reactor. A primary aim of this study was to differentiate the two main groups, as either “biologically bound” or “physicochemically bound,” with the quantification of bound phosphorus in animated sludge. After the four-level extraction treatment with distilled water and various solvents such as bicarbonate–dithionate, NaOH and HCl, the redox-sensitive-P and P bound to Fe(III)-hydroxides were found as main precipitation products in the anaerobic chamber (AN). Whereas the aerobic chamber (AE) contained mainly the precipitates of P bound to metals, such as Al and Fe, sorptive-bound phosphorus on metals and organic compounds etc. With iron precipitation, the levels of Fe–P, Al–P, and Fe–P adsorbed by Fe(OH)₃ were particularly high in the AE and AN. The sum of inorganic-P (physicochemically bound phosphorus) increased with iron precipitation, whereas that of organic-P and poly P (biologically bound phosphorus) decreased.

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1. Introduction

The elimination of phosphorus can occur not only through microbial metabolic procedures but also from precipitation reactions. With chemical precipitation, a material dissolved in the wastewater (here phosphorus) is transformed into an insoluble material by a chemical reaction with a precipitation agent. The insoluble material formed can be removed physically by settling out and/or filtration from the wastewater [1].

The phosphorus bound to the animated sludge can be classified into two main fractions, biologically bound and inorganically bound. The biologically bound fraction concerns assimilative bound phosphorus (phosphoric bio-molecules, e.g. DNA, RNA, ATP), whereas, inorganic polyphosphates represent the actual bio-P fraction (“luxury uptake effect”). Many studies have attempted to quantify this parliamentary group [2]. Physicochemical adsorption processes occurred basically either with van der Waals interactions of the adsorbate to the solid surfaces or the intrinsic or reciprocal effect and/or a covalent bonding with an organic substance. Here, the chemical bonding shows aluminum and potassium with the available concentration of phosphate in solution on the one hand and the dismantling of the polyphosphates in the microorganisms on the other hand. This specific adsorption behavior largely depends upon the environmental conditions (e.g. pH, Temp.).

The physicochemical bound fraction contains pure chemical precipitation products, such as calcium and iron phosphate. Phos-

phates can also be bound by adsorption to the activated sludge matrix [3]. An accurate classification of the types of elimination requires an examination of the phosphorus contained in the sludge with respect to the combined chemical and biological forms. With the P-fraction, a part of the polyphosphate is not considered to be stored intracellularly but is on the cell surface of bacteria [4]. Therefore, a method is needed to accurately differentiate between biologically coupled-phosphorus (organically coupled-phosphorus and inorganic poly P) and physicochemically coupled-phosphorus (P-precipitation products and adsorbed bound-P) in order to achieve a quantitative statement on the elimination mechanisms of biological phosphorus. Table 1 shows the summary of the best mechanisms to remove P-compounds at various extraction methods [5–8].

In this study, several forms of phosphorus in sewage were analyzed using a fraction method. The phosphorous content was classified into two main groups, “biologically bound” and “physicochemically bound,” the individual and/or bound phosphorus in animated sludge was quantified. This study may enable quantification of the soluble (SP) and adsorbed phosphorus (AP). In addition, the AP was sub-classified into inorganic and organic coupled-phosphorus (soluble reactive phosphorus (SRP) and non-reactive phosphorus (NRP), respectively). From these results, we can demonstrate an effective removal method for phosphorus from sewage as well as verifying elimination mechanism of phosphorus. The study was also conducted in separate aerobic and anaerobic chambers (AE and AN, respectively). Principal component analyses were carried out with 20 series of wastewater samples to provide a reliable basis for identifying the P-fraction obtained.

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Table 1
Summary of the best mechanisms to remove P-compounds at various extraction methods.

Methods	Best mechanisms for p-removal	Time required
Hot water-extraction Extraction by Weumberg and Orton [5]	Suitable for the extraction of polyphosphate Good for both hot water extraction and NaOH extraction to isolate polyphosphate	2 h (including measuring polyphosphate amount by acid hydrolysis) 4–5 h (including measuring polyphosphate amount by acid hydrolysis)
Extraction by Langen and Liss [6]	Good for measuring the amount of phosphate by cutting the polyphosphate chain using a hydrolysis reaction	ca. 10 h (including measuring polyphosphate by acid hydrolysis)
Extraction by Mino and Matsuo [7]	Differentiates metallic phosphate and low- and high-molecular polyphosphate	2 days, complex
Extraction by Psenner et al. [8]	Differentiate between chemical and biological connected phosphorus	3 days: (1st day: 8 h), (2nd and 3rd day: 2 h); complex

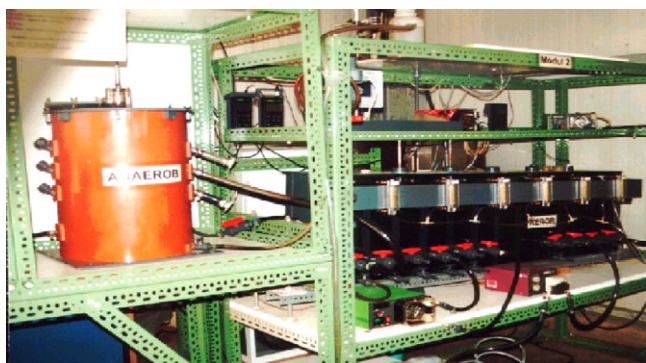


Fig. 1. The MBR (membrane bio-reactor)-laboratory plant.

2. Materials and methods

2.1. MBR-laboratory plant

A laboratory scale membrane reactor was used to analyze the results of biological phosphorus removal using a fractionation method. The MBR plant (Fig. 1) consisted of a cylindrical reactor and a cascade of 12 chambers. The volume of the plant (V_R) could be varied between 210 and 300 L. Each basin of the cascade could be operated in both ventilated and unventilated modes. In the storage vessel, the suspended and sediment materials were separated. Fig. 2 shows a schematic diagram of the MBR plant. The plant consists of four anoxic, three aerobic, and one anaerobic chamber with an external filter chamber. Wastewater initially flows to the anaerobic chambers, followed by three aerobic chambers, and is transported completely to the anoxic chambers (R_1). The wastewater is partly recirculated from the last anoxic chamber into the anaerobic chamber (R_2), and from the filter chamber into the first aerobic chamber (R_3). Excess sludge was removed from the fourth anoxic chamber. The water travels through the filter chamber, which allows the Q_{filtrate} to be collected. Table 2 shows the operating parameters of the MBR

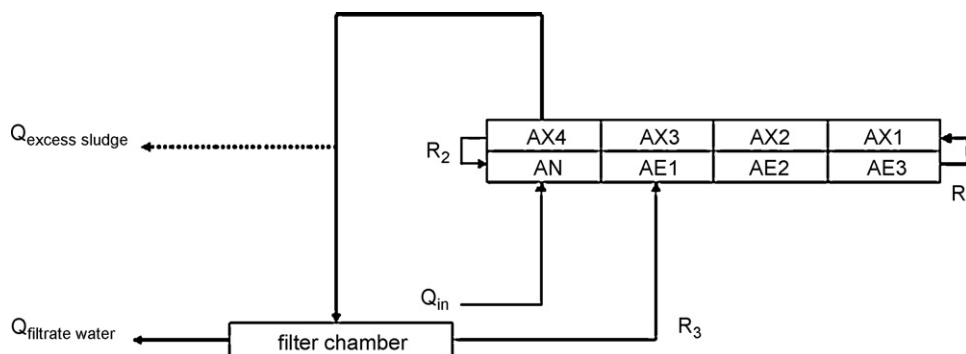


Fig. 2. Schematic diagram of the MBR-laboratory plant.

Table 2
Operating parameters of the MBR-laboratory plant.

Reactor	Volume (L)	SRT (h)	HRT (h)
Anaerobic (AN)	37	1.85	3.7
Aerobic (AE)	74	2.05	7.4
Filter chamber (FC)	(33)	(1.65)	
Anoxic (AX)	99	1.76	9.9
Total	210	–	21

Volume flow (net): 10 L/h.

Aerobic–anoxic R_1 : 10 L/h (100%).

Anoxic–anaerobic R_2 : 10 L/h (100%).

Sludge age: 15 days.

SRT: solids retention time; HRT: hydraulic retention time.

plant. The plant with a total HRT and sludge age of 21 h and 15 days, respectively, was prepared.

2.2. Sampling and analysis

The sample municipal wastewater was obtained from AN and AE of the MBR at Ruhleben, Berlin. The samples were taken from mid-May to November. For the filtration of aerobic samples, the remaining oxygen level was maintained at >2 mg O_2/L . Fractionation consisted of sequential extractions of the sludge samples with different extracting chemicals followed by incubation, centrifugation and analysis of the supernatant. In the supernatant, the TP and PO_4-P levels were determined after $0.45 \mu m$ filtration. The PO_4-P concentration represents the SRP. The difference between TP and PO_4-P is called the NRP. All samples were subjected to PO_4-P and TP analyses by ion chromatography with Dionex ISP 2000. TS and VSS were measured according to the DIN (German Institute for Standardization).

The solid samples after centrifugation were homogenized and treated with solvents such as bicarbonate–dithionate (BD), NaOH and HCl for the selective extraction of the phosphates bound to the solids. The individual extraction methods differed with respect

to the extracting reagents and number of extraction steps. The samples were subjected to the following fraction steps: detailed information of the steps and corresponding P-bindings were reported by Gonsiorczyk et al. [9] and Witt [4].

2.2.1. Step 0: PS-fraction (proven samples from sludge chamber)

A 25-mL raw sample was removed from the sludge container, and filtered under a pressure of 4 bar using a membrane filter (pore diameter 0.2 μm). The filter cake in semi-pellet form was removed from the filter and stored in a centrifuge glass for further fractionation. The filtrate was used to measure the dissolved phosphorus.

2.2.2. Step 1: DW-fraction (distilled water fraction)

The filter cake was taken into 25 mL distilled water, homogenized for 30 min using a magnetic stirrer, and subsequently centrifuged at 4800 rpm for further 30 min. The supernatant was decanted through a 0.45- μm membrane filter to measure the DW-SRP (soluble reactive phosphorus) and DW-NRP (non-reactive Phosphorus). The DW-SRP fraction denotes the loosely coupled or adsorbed phosphorus and DW-NRP indicates the loosely coupled or absorbed organic phosphorus, or poly P.

2.2.3. Step 2: BD-fraction (bicarbonate–dithionate fraction)

A pellet obtained from the centrifugation of DW-fraction in step 1 was suspended in 25 mL of the BD reagent (0.1 M), and the phosphorus was extracted for 30 min at 40 °C in an agitating water bath. After cooling, the mixture was centrifuged at 4800 rpm for 35 min. The supernatant was collected in a separate tube for analysis. This step was repeated and the decanted portions were placed in a beaker and subjected to a membrane filtration (0.45 μm). 25 mL of the filtrate was transferred to a 100-mL Erlenmeyer flask and filled with 1.25 mL of a 0.0025-M EDTA solution. The solution was purged with sodium dithionite moistened air for 10 min and used to measure the BD-SRP and BD-NRP. The corresponding P-fractions are as follows: BD-SRP, redox-sensitive-P mainly bound to Fe-hydroxides ($\text{FePO}_4(\text{Al})$, $\text{Fe}(\text{OH})_3\text{-P}$, $\text{Mn}(\text{OH})_4\text{-P}$); and BD-NRP, dissolved cell phosphorus.

2.2.4. Step 3: NaOH-fraction (sodium hydroxide fraction)

After step 2, the remaining solid was added to 25 mL of a 1-M NaOH solution and the phosphorus was extracted for 20 h at 25 °C in an agitating water bath. After 1 h, the mixture was filtered through a membrane filter (0.45 μm), and the liquid was used to analyze the fractions. The bound forms of the NaOH-fractions are as follows: NaOH-SRP, P bound to metal oxides mainly of Al and Fe (Al- and Fe-P, Al- and Fe-hydroxide); and NaOH-NRP, organically bound-P, e.g. P in microorganisms, detritus, humic compounds, poly P, P-lipid.

2.2.5. Step 4: HCl-fraction (hydrochloric acid fraction)

The solid obtained from step 3 was suspended in a 0.5-M HCl solution for 20 h at 25 °C in an agitating water bath. The liquid from membrane filtration (0.45 μm) was subjected to analyses of the HCl-fractions. The P-binding for HCl-SRP is carbonate- and apatite-bound phosphorus (Ca-P). For HCl-NRP, the P-binding is organic phosphorus or poly P. The solid remaining after the HCl-fraction had been finally suspended in 25 mL distilled water and the phosphorus content was measured to determine the total amount of phosphorus (TP).

2.3. Interpretation of the fraction method

The SP form was measured after digesting the filtrate. The resulting solution should contain all the filterable forms of phosphorus, both organic and inorganic, which have been converted to orthophosphate during the digestion process. However, the amount of phosphorus in this filterable pool is strongly dependent on the

filter used. With a larger the effective pore size of the filter, more particulate material will pass through the filter, become digested, and be considered “dissolved” [10]. Typically, a 0.45- μm cellulose filter was used as the standard. This filter excludes most particulates but colloidal phosphorus may still be present in the filtered fraction [8].

The SRP fraction should consist largely of the inorganic orthophosphate (PO_4) form of phosphorus. Previously, SRP is known as “soluble inorganic phosphorus.” This terminology was changed to “soluble reactive phosphorus” to reflect a more realistic interpretation of the forms of phosphorus found in this fraction. The terms “soluble” and “reactive” were chosen because this form of filtered phosphorus is not necessarily dissolved or inorganic. The term “reactive” is used to indicate that the phosphorus in the SRP fraction is not solely inorganic phosphorus but can contain any form of phosphorus, including some organic forms, which can react with the reagents. Some organic forms apparently hydrolyse and react. There is continuing debate as to the extent that SRP solely represents of the orthophosphorous form and its biological availability [11,12].

The NRP fraction contains filterable phosphorous forms that do not react quickly with the phosphorous reagents under the test conditions. The NRP fraction is measured as the difference between the total phosphorus and the SRP. The compounds in the NRP fraction are the organic forms of phosphorus, with the chains of inorganic phosphorous molecules, known as polyphosphates [13].

3. Results and discussion

3.1. Bound-P in each fraction

The coupled-phosphorus obtained from the four-level extraction treatment was determined almost exclusively from the BD- and NaOH-fraction. Fig. 3 shows the results of the direct distribution of each extract in the AE and AN. The SRP portion of the chilled water extract (DW extract) has been accepted as loosely adsorbed P. The portion of extractable phosphorus obtained from this step was quite small. This is to be reported that the NaOH and BD extracted fractions contain primarily the Al-P, Fe-P or organically bound phosphorous. In this study the BD- and NaOH-fractions contained ca. 90–95% of the phosphorous in the AE and AN, which has obtained prior to the HCl-fraction step, confirmed the phosphorous exists in sewage mostly in the form of bound phosphorous, i.e. either with Fe, Al or even organically bound. Similar results also reported previously for their related studies [4,8,9]. The bicarbonate dithionite extract (BD-P) is accepted as bound phosphate, with regard to the redox-sensitive SRP portion, i.e. Fe(III)-P-precipitation products and/or adsorbed to Fe(III)-hydroxides [14]. There was little iron released from the BD extract, which resulted from the transition of the trivalent iron phosphate to the bivalent form. The SRP in the BD extract was distributed equally in the AE and AN. However, the BD-NRP in the AN was substantially lower than the amount in AE. The NaOH-P content is defined by the status of the oxide-hydroxide-clay group and humic substances. The NaOH-P fraction should consist strictly of P-species associated with Fe-oxide/hydroxides, which are liberated during extraction by exchanging with OH-ions from the NaOH reagent. However, any P-substrate association that is susceptible to such exchange reactions, including P-organic compounds or associations, should contribute to the NaOH-P fraction [15]. The NaOH-extract is considered to be the SRP portion, and contains the chemically bound iron and aluminum phosphates. Most of the sludge P shifted into Ca-P and Fe-P fractions while that of the inorganic-P enters into soluble and Al-P fractions. Higher soluble P was observed at pH 7.0, Al-P and Fe-P at pH 5.8, and Ca-P at pH 6.4 and 7.0.

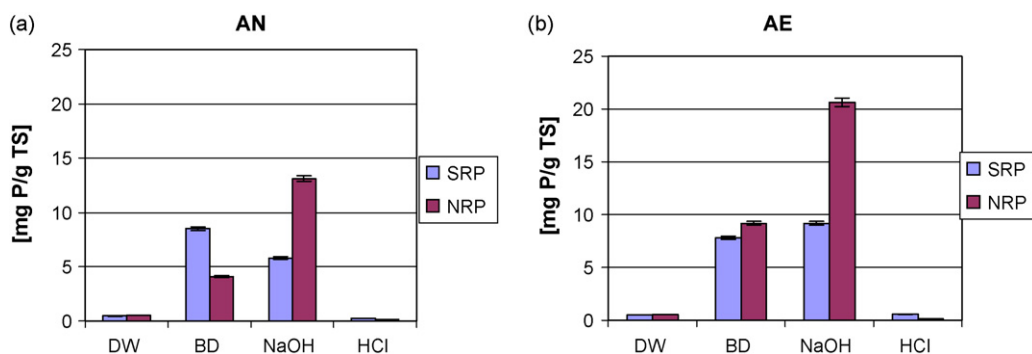


Fig. 3. Fractions in the (a) anaerobic (AN) and (b) aerobic (AE) chambers without iron precipitation.

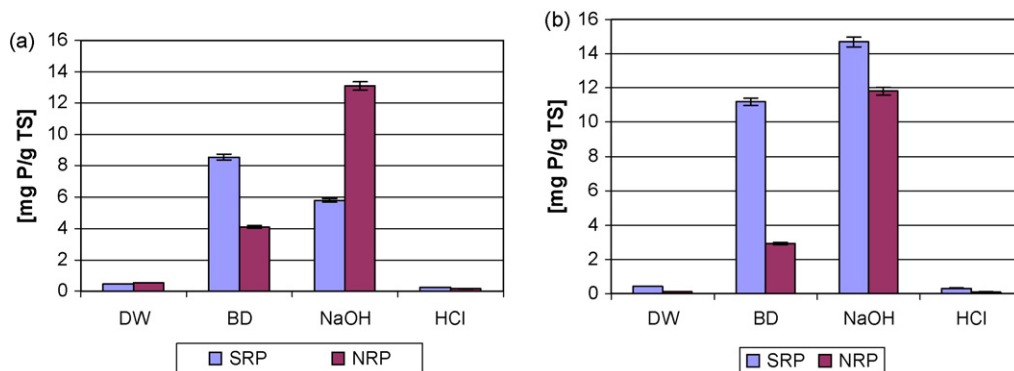


Fig. 4. Fractions in the anaerobic chamber (a) without and (b) with iron precipitation.

During the investigations, the amount of NaOH–SRP in AN was less than that of the BD–SRP (BD 8.54 mg/g TS and NaOH 5.8 mg/g TS). However, the results were opposite in AE (BD 7.83 mg/g TS and NaOH 9.2 mg/g TS). The phosphorus from the NaOH-fraction increased considerably under aerobic conditions (especially NaOH–NRP). The NaOH–NRP in the AN and AE were determined to be 13.1 and 20.63 mg/g TS, respectively. The portion of NRP phosphorus was greater in the NaOH than the BD-fraction.

Fig. 4 shows the results of each fraction in the AN with and without iron precipitation. The figure shows similar trends for each fraction in the AE. It means that NaOH–SRP was observed with iron addition while NaOH–NRP was obtained without iron as main precipitation. During the test for the precipitation of phosphorus, 3 mg/L of Fe(III) chloride (FeCl_3) was added to the influent. The dissolved inorganic phosphate (PO_4) precipitates with the application of iron or aluminum salts. However, this is partly removed from

the wastewater through physical flocculation processes, where it is adsorbed onto the precipitation products [10]. From the precipitation, the Fe^{3+} - or Al^{3+} -cation reacts with an anion, e.g. PO_4^{3-} . The quantity of phosphate bound to iron increased after adding FeCl_3 . The amounts of BD–SRP without precipitation were 8.54 mg/g TS (AN) and 9.2 mg/g TS (AE). On the other hand, with precipitation, these values were 11.19 mg/g TS (AN) and 11.3 mg/g TS (AE). The amounts of NaOH–SRP without precipitation were 5.8 mg/g TS (AN) and 9.2 mg/g TS (AE). In contrast, the amounts measured with precipitation were 14.59 mg/g TS (AN) and 19.14 mg/g TS (AE). The portion of SRP phosphorus in the NaOH–SRP appears to be much lower than the BD extract without precipitation. However, the results were reversed after precipitation. The trace amount of extractable phosphorus was obtained from HCl step. The P-fraction was sensitive to low pH in this step, and consisted mainly of apatite-P, P bound to carbonates and traces of hydrolysable organic-P. The

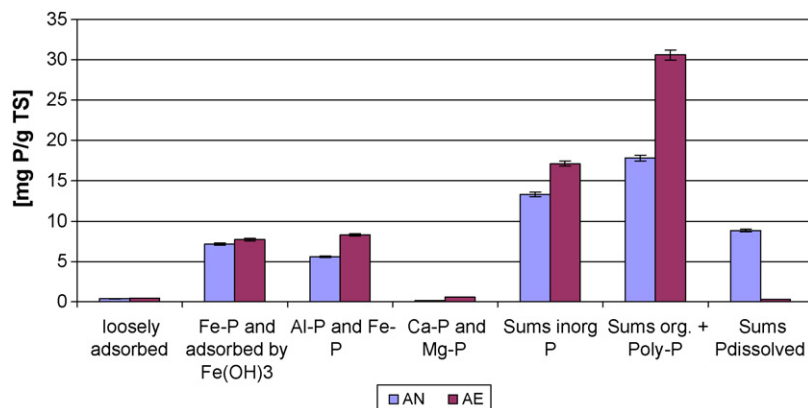


Fig. 5. P-coupled distribution without iron precipitation.

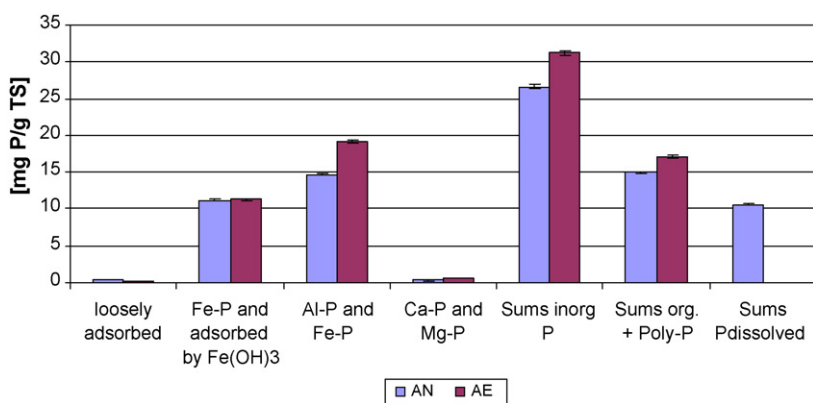


Fig. 6. P-coupled distribution with iron precipitation.

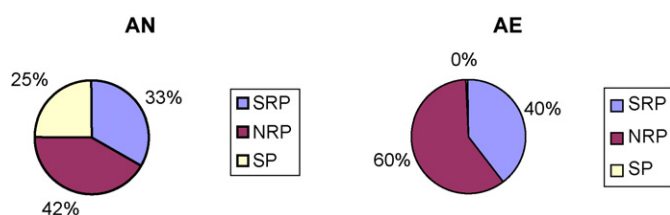


Fig. 7. P distribution in the anaerobic (AN) and aerobic (AE) chambers without iron precipitation.

Ca-P fraction was relatively stable, which was attributed to the permanent burial of P in sediments [9,16,17].

3.2. P-distribution

In dilute aqueous solution, phosphate exists in four forms. In strongly basic conditions, the phosphate ion (PO_4^{3-}) predominates, whereas in weakly basic conditions, the hydrogen phosphate ion (HPO_4^{2-}) is prevalent. In weakly acid conditions, the dihydrogen phosphate ion (H_2PO_4^-) is most common. In strongly acid conditions, aqueous phosphoric acid (H_3PO_4) is the main form.

Figs. 5 and 6 show the average P-bound distribution in the plant, both with and without iron precipitation, respectively. In this attempt with municipal wastewater, it was found that the amounts of loosely adsorbed-P, Ca-P and Mg-P were equally low with and without precipitation. There was little difference in the amount of the aluminum- and iron-bound phosphate between the samples with and without precipitation. With precipitation, Fe-P and Al-P, and Fe-P adsorbed by $\text{Fe}(\text{OH})_3$ were particularly high in both AE and AN. The sum of inorganic-P increased with precipitation, whereas that of organic-P and poly P increased without precipitation. Fig. 7 shows the distribution of phosphorus in the AE and AN without iron precipitation. During the investigations in both chambers, the amount of SRP was lower than that of NRP. The portion of NRP in AE was 20% higher than that of SRP, while the difference was approximately 10% in AN. The SP portion was negligible in AE. Fig. 8 shows

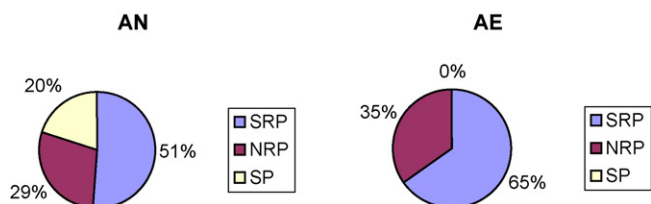


Fig. 8. P distribution in the anaerobic (AN) and aerobic (AE) chambers with iron precipitation.

Table 3

TP content in the laboratory plant (w/o Fe(III) chloride precipitation, $n=10$; w/precipitation, $n=10$).

	AN	AE
w/o precipitation	2.87% (1.68–2.98%)	3.28% (2.69–3.76%)
w/precipitation	2.25% (2.02–2.47%)	3.26% (3.02–3.5%)

the results of the same study with precipitation. In contrast to the case without precipitation, the SRP portion in the AN and AE was 51% and 65%, respectively. Compared with the values determined without precipitation, the portion of SRP in the AN and AE was increased by 22% and 30%, respectively. The portion of SP remained almost constant and was independent of iron precipitation.

3.3. Total P-content

TP is largely defined according to how much phosphorus in its various forms will be oxidized into orthophosphate by a specific oxidant. Table 3 summarizes the results for the TP content in the plant. The TP content in the sludge averaged 2.25–2.87% in the AN with and without precipitation, whereas the value in the AE was 3.26–3.28%. A P-content for the TS (total solid) of ca. 2–6% has been reported [18]. Therefore, the average P-contents found in this study were in the middle range of the values reported in the literature.

The TS content is an indication of the biomass in the reactor. Therefore, it can be used to determine the enrichment of inorganic materials, such as precipitation products or heavy metals. The amount of phosphorus that can be biologically released and/or taken up increases with increasing amount of active biomass available in the batch reactor [19]. Microorganisms will settle down

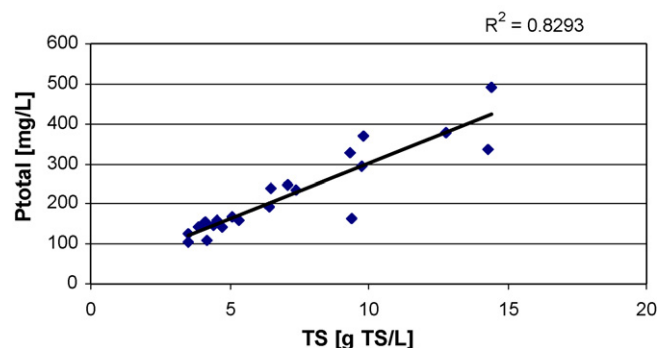


Fig. 9. The relationship between P_{total} and total solids (TS).

according to the sludge age and/or sludge load in a system. This is dependent on the concentration with equilibrium between the formed and removed biomass being reestablished. TS and/or its organic portion of TS are criteria well suited for such an evaluation. Fig. 9 shows the relationship between the TP and TS contents ($R^2 = 0.8293$).

4. Conclusions

In this study, several forms of phosphorus in municipal wastewater were analyzed using a fraction method. With iron precipitation, there was a decrease in the amount of biologically bound phosphorus in the municipal wastewater and an increase in the amount of physicochemically bound phosphorus in both the AN and AE. In particular, the quantity of biologically and physicochemically bound phosphorus was higher in the AE than in the AN. This might be due to the fact that aerobic microorganisms take up excessive amounts of TP in the AE.

Without precipitation, redox-sensitive-P and P mainly bound to Fe-hydroxides were the main precipitation partners in the AN, and P bound to metal, such as Al and Fe, and sorptive-bound-P on metals and organic compounds was the main precipitation partner in the AE. The main link between the phosphate in the AN and AE with precipitation was the P bound to metal oxides, which consist mainly of Al and Fe, sorptive-bound-P on metals and organic compounds. The calculated Ca-P and Mg-P were significantly low value in both chambers. The quantity of SP, with and without precipitation, was also similar, i.e. iron precipitation did not affect the soluble portion. Overall, the mechanisms of phosphorus in the municipal wastewater at Ruhleben in Berlin were determined to be BD-P and NaOH-P (90–95%) in the AE and AN, respectively.

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