

Article

Development of a Self-Sustaining Wastewater Treatment with Phosphorus Recovery for Small Rural Settlements

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Abstract: Global trends such as climate change and the scarcity of sustainable raw materials require adaptive, more flexible and resource-saving wastewater infrastructures for rural areas. Since 2018, in the community Reinighof, an isolated site in the countryside of Rhineland Palatinate (Germany), an autarkic, decentralized wastewater treatment and phosphorus recovery concept has been developed, implemented and tested. While feces are composted, an easy-to-operate system for producing struvite as a mineral fertilizer was developed and installed to recover phosphorus from urine. The nitrogen-containing supernatant of this process stage is treated in a special soil filter and afterwards discharged to a constructed wetland for grey water treatment, followed by an evaporation pond. To recover more than 90% of the phosphorus contained in the urine, the influence of the magnesium source, the dosing strategy, the molar ratio of Mg:P and the reaction and sedimentation time were investigated. The results show that, with a long reaction time of 1.5 h and a molar ratio of Mg:P above 1.3, constraints concerning magnesium source can be overcome and a stable process can be achieved even under varying boundary conditions. Within the special soil filter, the high ammonium nitrogen concentrations of over 3000 mg/L in the supernatant of the struvite reactor were considerably reduced. In the effluent of the following constructed wetland for grey water treatment, the ammonium-nitrogen concentrations were below 1 mg/L. This resource efficient decentralized wastewater treatment is self-sufficient, produces valuable fertilizer and does not need a centralized wastewater system as back up. It has high potential to be transferred to other rural communities.

Keywords: resource-oriented sanitation; phosphorus recovery; struvite precipitation; urine supernatant treatment



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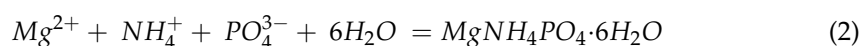
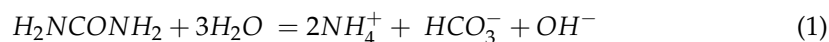
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1. Introduction

Rural areas need special solutions for sanitation as the connection to central sewer networks and wastewater treatment plants are expensive and the transport via trucks is not sustainable in terms of energy consumption and greenhouse gas emissions. On the other hand, decentralized wastewater treatment offers new solutions, including near-natural technologies and recovery of nutrients. Referring to the basic idea of resource-oriented sanitation (ROS), wastewater streams can be treated and resources recovered according to their specific properties starting with material flow separation at household level. The treatment of the grey water can be carried out with less effort and simpler technologies than the treatment of domestic sewage. Using separate collection of black water, or even better of urine and feces, the nutrients are available in a much higher concentration at a lower volume, which notably improves the possibilities for nutrient recycling. Struvite precipitation is one often investigated option to recover mainly phosphorus from urine as a valuable fertilizer. However, during this process, nitrogen is not completely recovered, leading to high concentrations in the supernatant, which need to be treated. In several

pilot or full-scale projects (e.g., [1–3]), the urine supernatant after struvite precipitation was discharged into a central WWTP or not addressed at all.

Urine can be used for phosphorus (P)-recovery via struvite ($MgNH_4PO_4 \cdot 6H_2O$) precipitation. Struvite is a slow-release solid fertilizer [4] which is easy to store and free from most organic micropollutants [5,6]. During storage of urine, urea is spontaneously hydrolyzed into hydrogen carbonate, hydroxide and ammonium (Equation (1)), leading to a pH shift from around 6 to ca. 9 [7,8]. This pH is convenient for struvite precipitation [9] so that only a magnesium (Mg) source needs to be added (Equation (2)). Due to its simplicity, this process has been considered one of the most suitable technology for P-recovery from urine [10].



Nevertheless, the process is influenced by several boundary conditions such as magnesium source, Mg:P ratio, stirring speed, reaction and sedimentation time, which have been investigated in several studies. As one of the most common magnesium sources, magnesium oxide (MgO) has the following advantages: the addition of magnesium oxide promotes the struvite precipitation reaction because of the increase in pH and the salting of the wastewater is low. However, magnesium oxide is a poorly soluble solid, and, compared to $MgCl_2$ and $MgSO_4$, MgO dissolves more slowly in urine [11]. A pre-treatment of MgO with water in suspension was used in a study of struvite precipitation in synthetic biologically treated swine wastewater by Capdevielle et al. [12], but not yet used in struvite precipitation in urine. The study by Krähenbühl et al. [13] indicates that the character of MgO crystals plays a role in the solution behavior in urine. The combined effect of the MgO type and the addition strategy has received less attention. Struvite has a molar ratio of $Mg:NH_4:PO_4$ of 1:1:1. As ammonium has a high surplus and magnesium is limited, magnesium needs to be dosed based on the concentration of phosphate. The dosage of magnesium differs in the form of Mg:P molar ratio from 1.0 to 2.0 in practical operations [1–3,14–17] of struvite precipitation in separated urine. In some of the above-mentioned cases, P-recovery reached more than 90% or even about 95% with a Mg:P molar ratio of 1.1–1.6. However, in studies by Dalecha et al. [1], the phosphorus recovery from urine achieved only 67% with Mg:P molar ratios of 1.5 and 1.8. The effect of stirring time was less investigated, while the MAP precipitation reaction takes place quickly and is completed within a few minutes in laboratory experiment [18]. In the literature, stirring time is fixed in most applications of struvite precipitation from urine and takes a maximum of 30 min: Etter et al. [3] used 10 min; VUNA [17], Winker [14] and Dalecha, et al. [1] used 15 min; and Morales et al. [2] and Antonini et al. [16] used 30 min after adding magnesium oxide. In studies by Dalecha et al. [1] and some of those by Winker and Rieck [14], the phosphorus recovery achieved only 65% and 67%, which is lower than the P-recovery in the study of Etter et al. (>90%) [3], although Etter et al. [3] used a shorter stirring time. The effect of stirring time should be discussed in connection with other operation factors, such as type and dosage strategy of magnesium oxide as well as the Mg:P ratio.

Full-scale struvite reactors have been developed for central treatment in municipal wastewater treatment plants [10,19] and are correspondingly technically complex with instrumentation, control and automation devices to control the process conditions. This is not suitable for decentralized units operated by non-experts in the field of water and chemical engineering. Hence, a good solution has to be found to set up a stable but simple process, without great measurement and control effort. It is therefore important to record fluctuations in the urine composition and find suitable operation conditions in order to make standardized specifications for the Mg dosage.

If the struvite precipitation process runs properly, phosphorus can be recovered almost completely, but there is still a large proportion of nitrogen remaining in the supernatant, which might compromise waterbodies if not removed. Hence, Antonini et al. [16] investigated a possible solution in pilot-scale for a further treatment of urine supernatant after

struvite precipitation by ammonium stripping and absorption. Therefore, the nitrogen-rich supernatant was heated to 40 °C and the pH was raised to 10 by adding sodium hydroxide, transferred to a stripper column to convert ammonium to the gas phase, followed by an absorption column with sulfuric acid. The end product ammonium sulfate can be used as a liquid fertilizer. A similar treatment of nitrogen-rich urine supernatant by stripping and acid adsorption was developed by Wei et al. [5] in pilot-scale and by Tao et al. [20] in lab-scale. Although nitrogen recovery is possible with these processes, an appropriate power supply and addition of chemicals (NaOH and H₂SO₄) is necessary, which are hazardous materials and have special requirements for storage and handling. Hence, there are no technically mature systems for nutrient recovery and waste material treatment available for urine that can be used for the decentralized area under comparatively unfavorable conditions such as limited supply of electricity. Bruch et al. [21] reported an improvement on the nitrification by using lava sand in vertical flow constructed wetland treating municipal wastewater. Within a long-term research, they showed that the average NH₄-N cleaning performance was 99% over 6.5 years, at a feed concentration of approximately 25 mg/L NH₄-N [22]. One reason for this high cleaning performance could be the sizeable specific inner surface area of the lava sand pores. However, whether such a filter can reduce ammonium at inflow concentrations of several 1000 mg/L has not been investigated yet.

The aim of this research was to develop an effective and stable P-recovery process as a part of a self-sustaining, small community wastewater system. The effects of operating parameters on P-recovery via struvite precipitation were investigated to find a standardized and easy operation mode for local people.

2. Materials and Methods

2.1. Development and Implementation of the Decentralized Wastewater Treatment and Resource Recovery System

The new, decentralized and self-sustaining wastewater treatment and resource recovery system was implemented in Reinighof, an isolated location in the countryside of Rhineland Palatinate (Germany). About ten people of different gender and generations live permanently together. Furthermore, a camping ground and seminar facilities belong to Reinighof, which lead to day peaks of up to 80 people on single days. Consequently, over a period of weeks and month, a mean value of 16 people per day was chosen as design value for the peak season. Before starting the project, Reinighof had already achieved self-sufficiency for water supply with its own spring and for power with a photovoltaic system. As no connection to a central wastewater treatment plant and no receiving water exists, the wastewater needed to be transported several kilometers via truck to a central wastewater treatment plant. Reinighof thus offers typical framework conditions for rural area: less stable and limited power supply, hardly any wastewater disposal infrastructure and local workforce not having special technical expertise for running their own facilities.

Since 2018, an autarky, decentralized wastewater treatment and resource recovery concept in Reinighof has been developed (see Figure 1) and tested. Three flows are separated: grey water from shower, handwashing and kitchen and urine and feces from urine-diverting dry toilets, which save water and energy. Constructed wetlands for grey water treatment have shown preference in practical application [23–25] due to their low operational costs and little need for technical expertise of the operational staff, which conforms to the challenge in Reinighof. Grey water is derived and pre-treated by sedimentation in a multi-compartment-shaft (hereafter, central shaft), from where the pre-treated grey water is fed without external energy by a surge feeder to a constructed wetland. The collect shaft and constructed wetland are dimensioned according the German guideline DWA-A 262 [26] for 16 people. Since Reinighof is located in a groundwater protective area, an outflow-free operation is decisive and implemented through an evaporation pond. As the composting process of human feces has been commonly used in ROS systems (e.g., [27–29]), a closed rapid composting with subsequent open post-composting is provided, whereby disinfection needs to be achieved so that the compost can be used as a soil conditioner and nutrient supplier on the property of the owner.

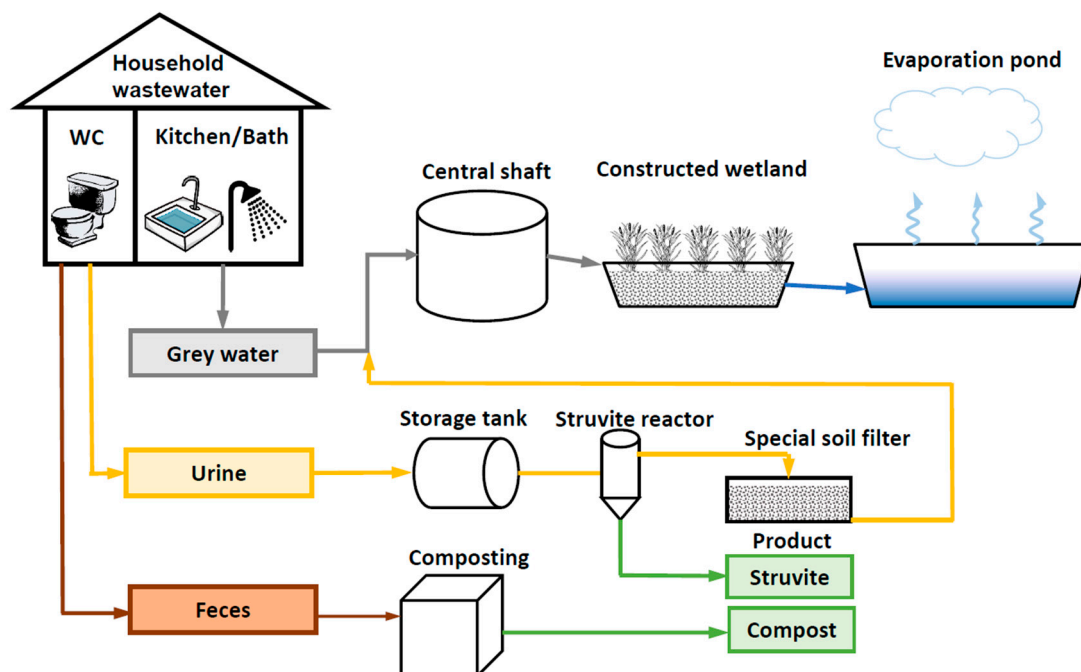


Figure 1. Concept of wastewater treatment and resource recovery system at the Reinighof.

The separately collected urine is discharged into a storage tank stored urine is pumped to a struvite reactor where struvite precipitation takes place by adding magnesium oxide (see Section 2.2). The ammonium-rich supernatant flows into a vertical flow special soil filter containing zeolite filter material, in which the ammonium is adsorbed and partially converted by nitrification (see Section 2.3). The outflow of the special soil filter is mixed with the collected grey water in the collect shaft and further treated in the constructed wetland and evaporation pond.

2.2. P-Recovery by Struvite Precipitation from Stored Urine

Fresh urine was stored in a polythene (PE) tank with 700 L volume, which can store the urine of 16 people for around four weeks. Every one or two weeks, 160 L of stored urine was pumped to a struvite reactor with a volume of 180 L. The stainless-steel struvite reactor consists of a cylinder drum and a conical bottom, with an electrical stirrer. After stored urine was pumped from the storage tank into the struvite reactor with running stirrer, a sample of stored urine was taken, and the phosphate concentration was analyzed immediately. The dosage of magnesium oxide is calculated and weighed depending on the chosen β value in each experimental series according to Equation (3). Afterwards, magnesium oxide was added by hand, via a vent of the top cover according to a molar ratio of Mg:P (β -value) from 1.0 to 2.0 for the different experiments. After finishing the magnesium oxide addition, samples are taken every 5–30 min from the struvite reactor, and immediately the phosphate-phosphorus concentration was analyzed. When the phosphate-phosphorus concentration reached <5 mg P/L or changed only slightly (<10 mg/(L* h)), the stirrer was turned off. The stirring time was recorded to find the suitable mixing time for the reaction and defined as befitting stirring time in this study. The precipitate was settled for 16–24 h, then collected in a filter bag and air-dried while the supernatant was led to the special soil filter after a urine supernatant sample was taken.

$$m_{\text{MgO}} = C_{p, \text{ stored urine}} * \frac{40 \text{ mg MgO/mmol}}{31 \text{ mg P/mmol}} * V_{\text{filling reactor}} * \beta \quad (3)$$

To find suitable operation parameters for a standardized easy operation mode and develop an effective and stable P-recovery process, 11 experimental series with in sum

28 runs with at least one repetition each were conducted (see Table 1). The effect of magnesium oxide type was tested by using three different MgO sources: G. MgO from Grüssing GmbH (Filsun, Germany) with 97% purity for technical usage; C. MgO from Carl Roth GmbH (Karlsruhe, Germany) with 98% purity for laboratory analysis; and W. MgO from Werthe-Metall (Erfurt, Germany) with 99.9% purity and manufacturer reference of MgO Light. To evaluate the influence of the dosage strategy, three different approaches were conducted: powdery magnesium oxide was added slowly for 7 min (hereafter, poud. sl.); 80–160 g MgO was first suspended in 0.8–1.6 L tap water for 10 min before the MgO suspension was added slowly for 7 min (hereafter, susp. sl.); or the suspension was added faster within 1 min or less (hereafter, susp. fa.). In other test series, the Mg:P ratio (β -value of 1.0, 1.3, 1.5 and 2.0) was varied as well as the stirring speed (78, 118 and 140 rpm).

Table 1. Operation conditions during the experiment.

ExperimentSet	MgO Type	MgO Adding Strategy	Molar Ratio of Mg:P (β)	Stirring Speed rpm	Repeat
1	G. MgO	poud. sl.	2.0	78	2
2	G. MgO	susp. sl.	2.0	78	3
3	G. MgO	susp. fa.	2.0	78	3
4	G. MgO	susp. fa.	1.5	78	4
5	G. MgO	susp. fa.	1.3	78	3
6	G. MgO	susp. fa.	1.3	120	2
7	G. MgO	susp. fa.	1.3	56	2
8	R. MgO	susp. fa.	2.0	78	2
9	W. MgO	susp. fa.	2.0	78	2
10	W. MgO	susp. fa.	1.3	78	2
11	W. MgO	susp. fa.	1.0	78	2

2.3. Urine Supernatant Treatment

After struvite precipitation, the ammonium-rich supernatant from the struvite-reactor was led to the vertical flow special soil filter (SSF) with $4 \text{ m}^2 * 0.95 \text{ m}$ (surface*H), filled with 25 cm gravel (2–8 mm) and 75 cm lava sand (0–4 mm), which contains about 10% zeolite and a high Brunauer–Emmett–Teller specific inner surface area (BET) (80–100 m^2/g soil) [21,22]. From 45 to 200 min after feeding the SSF, an effluent sample was taken and the concentrations of total nitrogen, nitrite-nitrogen, nitrate-nitrogen and ammonium-nitrogen were determined, to draw conclusions on the nitrification and nitrogen elimination efficiency of the SSF. Ammonium removal was defined as:

$$NH_4 - N - \text{removal} = (NH_4 - N_{SSF \text{ inflow}} - NH_4 - N_{SSF \text{ outflow}}) / NH_4 - N_{SSF \text{ inflow}} * 100\% \quad (4)$$

Before and during the investigation phase, samples of the effluent of the constructed wetland (grey water treatment) were taken, once before setting the MAP reactor into operation (effluent quality without influence of the urine-treatment step) and afterwards every three months to understand to which extent the urine treatment influences the effluent quality of the grey water treatment.

2.4. Analytical Methods

Electrical conductivity and pH value were measured with a portable conductivity meter (WTW Cond 3110, Weilheim, Germany) and a portable pH meter (WTW pH 323, Germany). The concentrations of phosphate-phosphorus ($\text{PO}_4\text{-P}$), nitrate-nitrogen ($\text{NO}_3\text{-N}$) and nitrite-nitrogen ($\text{NO}_2\text{-N}$) were measured using a portable Hach DR 1900 photo spectrometer (Germany) with cuvette test (LCK 348 for 0.5–5.0 mg/L $\text{PO}_4\text{-P}$, LCK 339 for 0.23–13.5 mg/L $\text{NO}_3\text{-N}$ and LCK 342 for 0.6–6.0 mg/L $\text{NO}_2\text{-N}$). Ammonium-nitrogen ($\text{NH}_4\text{-N}$) magnesium (Mg^{2+}), calcium (Ca^{2+}), potassium (K^+) and sodium (Na^+) were analyzed with ion chromatography (Metrohm 930 Compact IC Flex, with column Metrosep RP 2 Guard, Swiss), while total nitrogen (N_{tot}) and total phosphorus (P_{tot}) were determined by standard methods (DIN_12260 and DIN_EN_ISO_6878). Analyses of N_{tot} and P_{tot}

were done directly from homogeneous samples, while the samples were filtered with a final pore size of 0.45 μm (Th. Geyer, Renningen, Germany) before measurement of ionic concentrations.

3. Results and Discussion

3.1. Composition of Stored Urine

Table 2 shows the composition of stored urine from the Reinighof (28 samples) in comparison to values from the literature [1,3,5,11,30–32]. In all samples, the pH reaches values around 9, which is in accordance with literature data, although fresh urine is conveyed daily into the urine tank. This pH is suitable for struvite precipitation, so that no addition of base is necessary. Phosphate-phosphorus concentrations are fluctuating between about 260 and 460 mg/L with a mean value of 335 mg/L, which is in the same range as found by Freguia et al. [30] and Wei et al. [5], but higher than concentrations described by Hug and Udert [32], Ramesh and Sakthivel [11] and Etter et al. [3]. Ammonium concentrations of stored urine in Reinighof are higher than those described by Hug and Udert [32], Ramesh and Sakthivel [11] and Etter et al. [3]. While the molar ratio of $\text{NH}_4:\text{PO}_4$ in the literature data vary from 28 to 52, the values of this study fluctuate only slightly between about 24 and 28.

Table 2. Physical and chemical characteristics of stored urine at Reinighof (28 samples) and average values or single values in literature.

		Temp.	Cond.	pH	$\text{PO}_4\text{-P}$	P_{tot}	$\text{NH}_4\text{-N}$	N_{tot}	Mg^{2+}	Ca^{2+}	K^+	Na^+
		$^{\circ}\text{C}$	mS/cm	-	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
In this study	average	13.2	31.3	9.2	335	382	4337	4833	1.1	8.5	1576	1764
	min.	6.8	25.6	9.0	264	266	3433	3525	0.0	4.9	1367	1457
	max.	19.0	36.3	9.4	457	484	5365	5855	2.8	18.8	1856	2026
	median	12.0	30.7	9.2	340	379	4243	4798	0.0	8.1	1561	1757
Dalecha et al. [1] ^a			28.8	8.6	293							
Etter et al. [3] ($n = 14$)			25.9	9.0	195							
Wei et al. [5] ^a				8.9	384		5615		<50		1166	2300
Sakthivel et al. [11] ^b			25.0	8.8		187	2720		1.8	17.7	1330	1670
Freguia et al. [30] ^a			45.0	8.8	330		7800			10.0	2400	2900
Zamora et al. [31] ^a				9.3	223		3700		0.9	17.0	1900	2400
Hug und Udert [32] ($n = 9$)			25.0	8.9	197		2540		1.6	16.5	1980	

^a n , unknown; ^b single value.

Dalecha et al. [1] and Etter et al. [3] suggested a correlation between conductivity and phosphorus concentration which could offer a simple detection method for determining the amount of magnesium which needs to be dosed. In both studies, conductivity and phosphorus concentration from ten samples of stored urine showed good correlation ($r^2 > 0.9$). Different from those studies, the correlation ratio of conductivity and phosphorus concentration of 28 samples in Reinighof is low (see Figure 2). Except the phosphate concentration, the concentrations of ammonium, potassium and sodium are very high (Table 2) in the stored urine, which affects conductivity, too. Particularly, the ammonium concentration is influenced by urea hydrolysis and thus changes with storage time while the P-concentration is constant, if no fresh urine is added. Hence, this method is not suitable in the case of Reinighof and presumably other facilities where the storage time is not constant. Therefore, another simple dosing strategy is needed. Although sporadically very high or low P concentrations occur, about 70% of the 28 samples show a quite stable concentration of 340 ± 30 mg/L (see Figure 2). Using a fixed magnesium amount per liter stored urine would consequently lead to only a slight over- or underdosage within the range of 10%. Based on these considerations, a strategy for magnesium dosage was developed, which is described in Section 3.2.

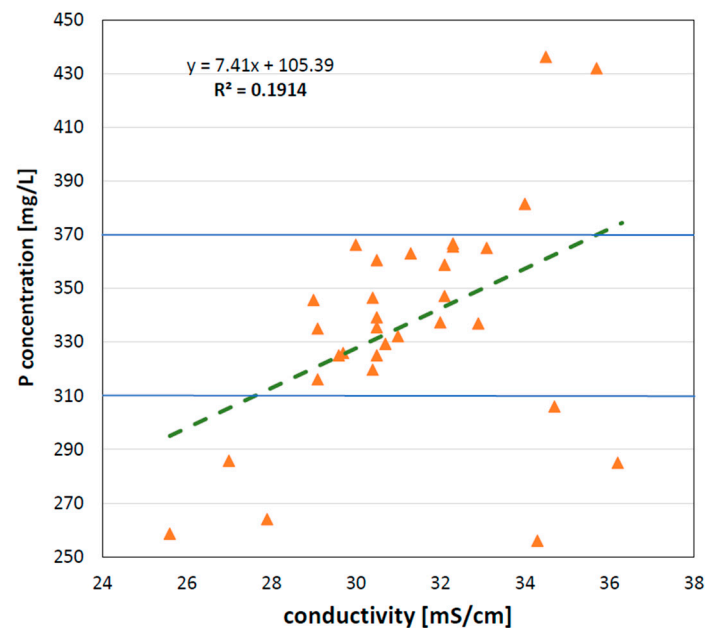


Figure 2. Correlation between conductivity and phosphor concentration of 28 stored urine samples in Reinighof. Seventy percent of the samples have P-concentrations within the two blue lines.

3.2. Phosphorus-Recovery via Struvite Precipitation

Figure 3 shows the phosphorus concentration in urine before adding magnesium oxide and in the supernatant after the reaction and sedimentation of the precipitate under different operational conditions. Additionally, the befitting stirring time is determined in each case. Generally, with a dosage of magnesium oxide within the molar ratio Mg:P of $\beta = 1.0$ – 2.0 , a P-recovery of over 85% could always be reached, yet mostly over 90% under befitting stirring time, which varied between 10 and 210 min. This differs somewhat significantly from the fixed stirring time in other application of struvite precipitation from urine, which took a maximum of 30 min [1–3,14–17] (see Section 1). The effect of the stirring time is discussed in connection with other operation conditions below.

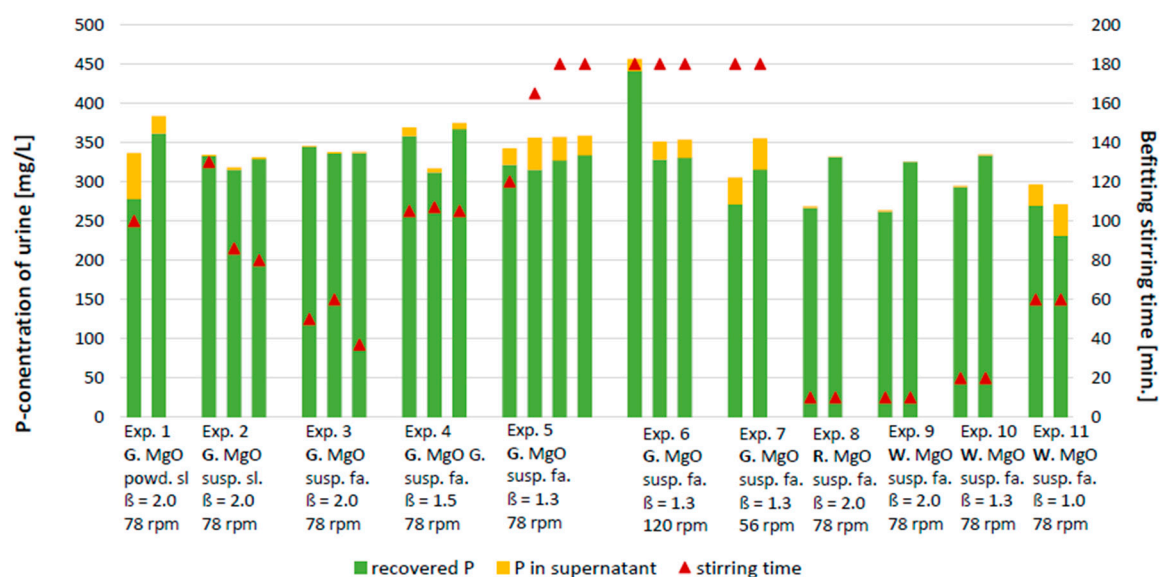


Figure 3. P-recovery in dependence of the stirring speed, Mg-dosing strategy and β -value. Phosphorus concentration in urine (whole column), in the supernatant after MAP precipitation (yellow part of the columns) and decline through precipitation (green column). Befitting stirring time in red triangle on secondary coordinates.

Experimental Sets 1–3 (Figure 3) were carried out under the same conditions except the MgO dosage strategy. The results show that, even after a longer stirring time, the P-recovery by adding MgO as powder (powd.sl) is less than by adding MgO in suspension (susp. fa. and susp. sl.). This is because magnesium oxide is a poorly soluble solid and dissolves slowly in urine [11]. However, a pre-treatment of MgO in suspension with water increased the precipitation velocity, which was also mentioned by Capdevielle et al. [12]. Stolzenburg et al. [33] indicated that adding MgO to water results in a dissolution step and a Mg(OH)₂ formation step, which leads to a predominance of Mg(OH)₂ on the surface of the suspended particles acting as a struvite precipitation precursor. Hence, a preparation of MgO suspension is suggested for a useful pretreatment for struvite precipitation from urine.

With decreasing $\beta\beta$ -values (2.0, 1.5 and 1.3) in Experiments 3–5 using suspended G. MgO and a fast dosage, the needed stirring time increased from 60 to 180 min while phosphorus recovery decreased from over 99% to 85%. Meanwhile, in Experiments 9–11 with W. MgO, the required stirring time increased with decreasing β -value, too. However, the reaction time was shorter than in the above-mentioned experiments with 60 min at $\beta\beta = 1.0$, 20 min at $\beta = 1.3$ and only 10 min at $\beta = 2.0$. The P-precipitation was less than 90% with $\beta = 1.0$ but more than 99% with $\beta = 1.3$ and 2.0. Overdosage of magnesium promotes the velocity of struvite precipitation and P-recovery. Winker and Rieck [14] reported that P-precipitation increased from 89% to 99% by increasing the β value from 1.0 to 1.4. They found that, with a β -factor over 1.4, a further increase in P-precipitation is hardly possible. This is in accordance with the experiments conducted in this study with the light W. MgO. However, using G. MgO, the results are different. This can be explained by a “loss” of active MgO due to the production of the suspension so that the effective $\beta\beta$ -value might be less than the calculated one leading to a further increase of the P-recovery with rising $\beta\beta$ -factor in the range of 1.3–2.0. This emphasizes that the selection of MgO plays an important role for the application. Experiments 3, 8 and 9 show that different types of MgO have different effects on the struvite precipitation process. This might explain lower P-recovery in the experiments of Daleccha et al. [1], too. Krähenbühl et al. [13] pointed out that the character of MgO crystals plays a role in the dissolution behavior in urine. Romero-Güiza et al. [19] reported a similar effect with different MgO and struvite precipitation after acidification from pig slurry.

The effect of stirring speed varying from 56 to 120 rpm was proved as irrelevant (Experiments 3, 6 and 7). Other authors [34] detected that the P-recovery increased slightly with stirring speed in range of 30–60 rpm, which is below the range investigated in this study.

Based on the above-mentioned results, suitable operation conditions were defined for the further operation of the struvite-reactor at the Reinighof:

- Selection of a light MgO, which needs to be pre-tested
- Fast addition in form of suspension
- Overdosage of MgO in the range of $\beta = 1.3$ –1.5
- Stirring speed 78 rpm and stirring time 1.5 h

For further operation of the reactor without regular analysis of the P-concentration, a fixed mass of MgO per volume is recommended. Table 3 shows that, depending on the measured phosphorus concentrations in the urine, different MgO amounts are needed to keep a constant Mg:P ratio. To ensure a ratio over 1 but below 2, a dosage of 100 g MgO can be suggested per filling with 160 L urine of the reactor, which means a Mg:P ratio between 1.2 and 1.9, referring to the maximum and minimum P-concentration measured in the urine, respectively, and a Mg:P ratio of 1.4 with average and median P-concentration. Within the 70% range of P-concentrations (see Figure 2), the Mg:P ratio fluctuates between 1.3 and 1.6, which comprises the above-defined best suitable β range of 1.3–1.5.

Table 3. Range of phosphorus-concentration in urine and Mg:P ratio at different amounts of MgO dosage.

Concentration of PO ₄ -P in Stored Urine (Mg/L) in 28 Samples (See Table 1)	Mg:P Ratio in Dependence of the Mass of MgO Per Filling with 160 L Urine					
	80 g	90 g	100 g	110 g	120 g	
min.	256	1.5	1.7	1.9	2.1	2.3
70%-lower average	310	1.3	1.4	1.6	1.7	2.0
median	335	1.2	1.3	1.4	1.6	1.7
70%-upper	340	1.2	1.3	1.4	1.6	1.7
max.	370	1.0	1.2	1.3	1.4	1.6
	436	0.9	1.1	1.2	1.2	1.3

3.3. Ammonium Removal in the Special Soil Filter

The elimination of ammonium in the special soil filter was 93.5% on average, although the mean inflow-concentrations were high with 4244 mg/L NH₄-N. Figure 4 demonstrates that, with start-up of the operation in November 2018, ammonium was retained totally and no nitrate was produced. This indicates adsorption of ammonium or ion-exchange in the soil due to the zeolite-containing lava sand and the high cation exchange capacity of the special lava sand (650 µmol/g in comparison to <30 µmol/g of other lava or fluviate sand) [21]. In mid-April 2019, the nitrogen retention dropped and the ammonium concentration rose slowly. With some time-delay, nitrate was produced via nitrification. First, the NO₃-N concentrations remained relatively low until mid-May but rose sharply from mid-June 2019 onwards, while the ammonium concentration dropped down. This indicates an improvement of the nitrification. From April to June, the retention of nitrogen dropped slowly. In July, there was a sharp drop, correlated with rising nitrate concentrations. This shows that the SSF is able to convert a high percentage of ammonium to nitrate and nitrite and to retain part of it. Original Data of Table 2 and Figures 2–4 in Supplementary Material.

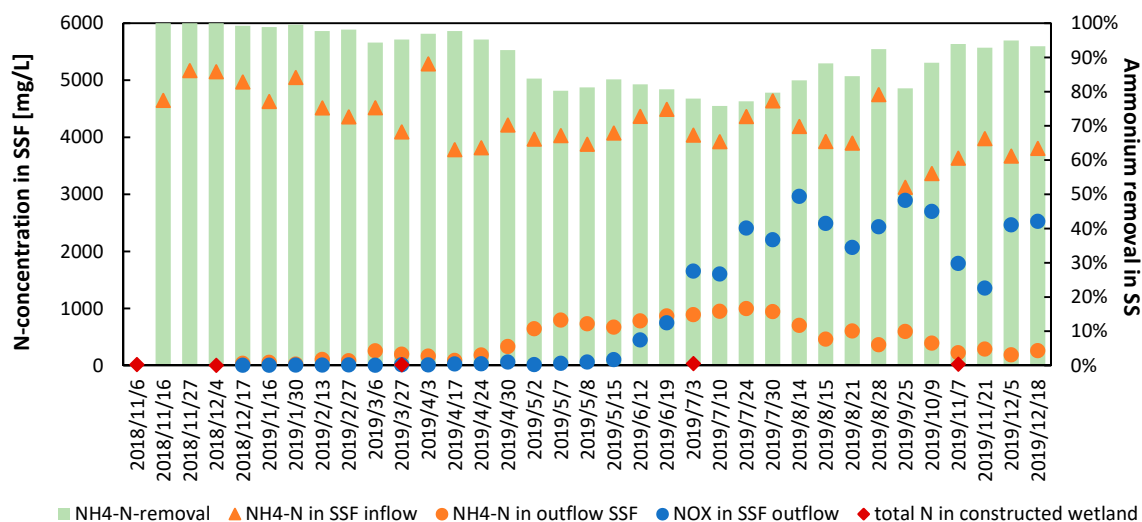


Figure 4. N-concentration in urine supernatant. SSF outflow and constructed wetland outflow (on major axis) and NH₄-N-Removal in SSF (light green column on secondary axis).

3.4. Effluent of the Constructed Wetland

Despite the high ammonium removal of 93.5% in the SSF, the N-concentrations in the effluent of the SSF are still high. NH₄-N-concentrations in maximum 995 mg/L and NO_x-N-concentration up to nearly 3000 mg/L are reached. This needs further treatment. Therefore, the effluent was pumped into the central shaft, pre-elimination of the grey water treatment was performed, and it was treated in the constructed wetland. The ammonium concentrations in the effluent of the constructed wetland were always below 1 mg/L NH₄-

N (see Table 4), which indicates a very high purification. Additionally, the effluent of the constructed wetland was surveyed sporadically by the municipality. In 2018 and 2019, the COD was below 20 mg/L and $\text{NH}_4\text{-N}$ below 1 mg/L.

Table 4. N- and P-concentration in the outflow of the constructed wetland for grey water treatment.

Date	N_{tot} mg/L	$\text{NH}_4\text{-N}$ mg/L	$\text{NO}_3\text{-N}$ mg/L	P_{tot} mg/L
2018-11-06 *	15.1	3.10	<0.06	0.70
2018-12-04	3.1	0.10	<0.06	0.44
2019-03-27	13.6	0.77	<0.06	0.10
2019-07-03	34.0	0.74	31.76	0.08
2019-11-07	20.9	0.08	20.20	0.12

* Before connection with urine-treatment.

4. Conclusions

The results show that a self-sufficient decentralized wastewater treatment and phosphorus recovery is possible by separation of grey water, urine and feces. Grey water was easily treated in a constructed wetland, while feces can be composted. Urine was treated in a new system for P-recovery, consisting of a urine storage tank, a struvite precipitation reactor and a special soil filter for the treatment of the ammonium rich supernatant with consequent treatment in the grey water treatment via constructed wetland. This resource-oriented sanitation system leads to an efficient phosphorus recovery and ammonium removal. After selecting the MgO source, the struvite precipitation reactor can be operated easily by local people without analytical devices and control efforts. The following tasks need to be done: pumping stored urine in a struvite reactor, suspending 100 g MgO in 1 L water and adding the suspension quickly into the struvite reactor. After 1.5 h stirring time and 16–24 h sedimentation time, precipitated struvite can be collected in a filter bag and air-dried. With those easy operation steps, over 90% of the phosphorus from urine can be recovered. The investigations showed that the ammonium rich supernatant of the struvite reactor can be treated with a vertical flow special soil filter and additional treatment with the grey water in a constructed wetland.

In the one-year investigation period, the ammonium concentrations in the effluent of the constructed wetland were always below 1 mg/L $\text{NH}_4\text{-N}$, which indicates a very high purification. Overall, resource efficient decentralized wastewater treatment for rural communities can be successfully implemented without having a centralized wastewater treatment as back up while integrating resource-efficiency and using synergistic effects of different treatment steps. This system has high potential to be transferred to other cases of small rural communities.

Supplementary Materials: The following are available online at <http://www.mdpi.com/xxx/s1>.

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Abbreviations

BET	Brunauer–Emmett–Teller specific inner surface area (m ² /g)
β	molar ratio Mg:P
Cond.	Conductivity (mS/cm)
Ca	calcium, concentration (mg/L)
K	potassium, concentration (mg/L)
Mg	Magnesium, concentration (mg/L)
MgO	Magnesium oxide
Na	sodium, concentration (mg/L)
NH ₄	Ammonium
NH ₄ -N-removal	Ammonium removal (%)
NH ₄ -N	Ammonium-nitrogen, concentration (mg/L)
NO ₃ -N	nitrate-nitrogen, concentration (mg/L)
NO ₂ -N	nitrite-nitrogen, concentration (mg/L)
N _{tot}	total nitrogen, concentration (mg/L)
P	phosphorus
PO ₄ -P	phosphate-phosphorus, concentration (mg/L)
P _{tot}	total phosphorus, concentration (mg/L)
ROS	resource-oriented sanitation
struvite	Magnesium-ammonium-phosphate, MgNH ₄ PO ₄ ·6H ₂ O
SSF	special soil filter
Temp.	Temperature [°C]

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