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Phosphorus consumption. From linear to circular flow

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Abstract

Wastewater and waste sludge are generated in all parts of the world during domestic and industrial activities. Conventional wastewater treatment methods generate a highly concentrated municipal sludge, which needs to be disposed of effectively without leading to secondary pollution. Animal manure and post generated manure wastewater are another environmental concern. Both of the above wastewater and sludge/manure are rich in organic/inorganic forms of carbon, nitrogen (N) and phosphorus (P). Anthropogenic and industrial activities in the global biogeochemical cycles have resulted in a drastic one-way mobilization of these resources into the atmosphere and the environment. The current intensive agriculture requires huge quantities of nitrogen (N) and Phosphorus (P) containing fertilizers. The industrial production of ammonia and nitrates is quite energy demanding; however, nitrogen is abundantly present in the nature and therefore it is a non-restricted resource for nitrogen derivate production. Unlike nitrogen, P can be obtained primarily from mineral deposits available only in few geographic locations. The phosphate rock reserves are finite and the current intensive fertilizer production based on economically mined rocks could last only another 50-100 years. Since phosphates are available only in limited geographic locations and the recognition that geologic phosphates are a non-renewable resource, Phosphorus recovery becomes a crucial for sustainable food production as EU depends for 90% on import of phosphate rocks (European Commission 2017). Within the EU only Finland has some phosphate rocks. The list of supplying countries is quite short; more than 70% of the present known global reserves of phosphate rock are located in Morocco as of all mined and processed phosphate rock (2009). Phosphorus, being a finite resource with deficits starting approximately from the year 2070 due to increased demand might also result in high prices and reliance on single point sources, giving them monopoly over the market. The EU phosphorus flows show that the main losses of phosphorus in the food sector are through sewage sludge, other waste water and food waste. In general, phosphorus can be recycled, mainly from wastewater (e.g. sewage water), manure and organic waste (e.g. wasted food). Thus, wastewater can be

considered as a renewable source of N and P. Instead of releasing the N and P rich wastewater into coastal and inland waters increasing eutrophication risk their utilization can be beneficial resulting in multiple benefits like nutrient recovery, water reclamation for reuse and maintenance of ecological balance in aquatic systems. Presently, the recovery and reuse of P is still far from being a main stream practice. Yet, the techniques already accepted and applied differ by the origin of the used matter (wastewater, sludge, ash) are mainly focused on the process of precipitation. One of these techniques is struvite precipitation, which can be implemented in wastewater treatment plants that use enhanced biological or semi biological/chemical phosphorus removal. Struvite (magnesium ammonium phosphate or MAP ($MgNH4PO4·6H2O$)) is formed by a basic precipitation reaction in different stages of the wastewater treatment process where magnesium (Mg^{2+}), ammonium ($NH4^{+}$) and orthophosphate ($PO4^{-3}$). The article discusses the progress in extracting P from sewage sludge and animal manure, the conditions to create optimal conditions for struvite precipitation in such media and the way to overcome the problems associated with choosing the right Mg source, pH adjustment and the non-acceptable level of organic matter in the initial suspension.

Keywords: *circular economy, P recovery.*

1. Introduction

Phosphorus importance and deficiency

Phosphorus (P) is an essential nutrient for all forms of life [1]. Specifically, P is important nutrient in agriculture, a major limiting factor for plant and the entire food production chain. Currently mineral fertilizers are irreplaceable part of modern agriculture. In natural ecosystems, P is entirely supplied from the weathering of parent materials [2], and the amount of total P is preserved because it is released back to the soil system through plant residues, animal excreta or when organisms die. In agricultural systems, crop removal represents the primary route by which P is lost from soils [3]**.** Phosphorus (P), is a finite resource obtained mainly from rocks located in few regions of the world [4]**.** Morocco, for instance, controls 75% of the remaining world's phosphorus reserves. This creates vulnerability to food production systems and increases the need to encourage sustainable phosphorus production practices [5]. P stocks steadily decrease with time [3]. Mineral phosphate fertilizers are the primary source of P input to agricultural lands [6]. Globally, phosphate fertilizer production resulted in P depletion is approximately 20 Mt of P coming from phosphate rock [7]. Of all mined and processed phosphate rock in 2009 (32.8 million tons of phosphorus) more than a third is lost during the mining and manufacturing process (11.0 MT), more than half is turned into fertilizers (17.5 MT), and some phosphate rock is used for additives in livestock feed (1.7 MT) and food (0.3 MT). In total 19.5 MT of phosphorus per year goes into the agricultural system Phosphorus rock supply in a baseline development is sufficient for the coming centuries, but the concentration of reserves and the almost complete dependence on imports of the EU and other regions in the world create geopolitical supply risks [8]. The demand for P is expected to increase in the following years due to continuous population growth and rising global demand for food, with a predicted considerable increase [9]. It has been estimated that the demand for mineral P fertilizer will increase in the coming decades as the global population is expected to increase by almost 50% and globally more people include meat in their diet [10]. Phosphate rock is on the EU list of critical raw materials. P rock is a finite, non-renewable resource. According to the U.S. Geological Survey, phosphate deposits will last about 50 years at the current rate of extraction [11]. Therefore, there is an increasing concern regarding phosphate rock reserves to become depleted. Recognizing that the *Phosphate rocks* a finite resource are of great importance as a major source of mineral fertilizers necessitates the need for identifying and usage of P rich waste streams, both municipal and agricultural. Part of the increased pressure on P resources could be alleviated by recycling P contained in various agricultural and urban wastes [6]. In the same time the P depletion and formation rate should be controlled efficiently as possible and recycling to be optimized. Currently, the actual use of P resources seems far from sustainable regarding all area of potential P sources -urban, industrial and agricultural waste. The need of knowledge of P status and the lack of a clear regulation organic waste management still limits P recycling potential in agriculture. All these necessity steps for better P recovery can change the current 'once-through' usage of P in the food production chain [12, 13]. towards a more sustainable utilization implying efficient mineral fertilizer use, minimizing P losses and more intensive utilization and recycling of animal manure and other secondary P-rich waste products. Van Dijk et al. [14] analyzed phosphorus flows for the EU27 in 2005. Agricultural land is fertilized with 1389 KT (kilotons) per year) of phosphorus, 1749 KT phosphorus from manure, and 157 KT phosphorus from other recycling. Accumulation has a negative impact on the environment in regions with excess supply of manure. Stricter fertilizer and manure regulation have been implemented to reduce the nutrient loss. The EU phosphorus flows [15] show that the main losses of phosphorus in the food sector are through sewage and other waste water and food waste. To reduce the inflow of phosphorus in the EU (1389 KT fertilizer, 249 KT feed additives, 189 KT plant-based animal feed and 27 KT inorganic food additives) these waste flows have to be recycled. Phosphorus in manure is almost completely recycled, while losses from the fields or from stables are relatively minor, but with a relatively high negative environmental impact. The policies implemented to prevent over-

fertilization from manure, show how policies influence recycling of phosphorus from manure, which may be an example for recycling of other parts of the phosphorus flows. Resource security is emphasised in a number of EU strategic documents, highlighting the need to promote measures to increase the efficiency of natural resource management by increasing the share of waste streams for reuse, avoiding losses, and intensifying material recycling. The "circular economy" is now a priority for the European Union's economic policy. The objective of sustainable management of natural resources is to reverse the negative trend of over-exploitation and to reduce losses [16].

2. Move to circularity

During the recent years the society builds the idea to move from the linear system of P extraction/processing and application of the produced fertilizers into applying a mechanism of circularity. Such a concept lays in the hearth of the Manifesto for a Resource-Efficient Europe, the European Commission acknowledged the inevitability of the transition toward a regenerative Circular Economy (CE). The concept of a CE involves steps towards avoidance of losses, which can be found all along the P supply chain in varying degrees of magnitude and leads to low nutrient-use efficiencies [17]. Obviously, the shift passes through application of multidimensional innovations in sense of knowledge development of products/processes characteristics, considering various perspectives including all technological and economic aspects. Such innovations should be applied particularly for the case of phosphorus, one of the major macronutrients representing a critical building element for today's fertilizer production and, thereby, for global food security [17]. Phosphorus is produced almost exclusively through the mining of finite phosphate rock deposits of either sedimentary (87%) or igneous (13%) origin. From the overall production volume, 83% (46 Mt P_2O_5) is used for chemical fertilizers and the rest is used for other industrial purposes [15]. The currently mostly linear P supply chain includes exploratory activities, extraction (mostly open-pit), product enrichment, processing of various fertilizers, and consumption in the form of phosphate fertilizers. Once applied as a fertilizer, P is taken up by plants. The rest remains as a stock in the soil from where parts are leached into our aquatic system and become sediments. End-of-pipe solutions are used by recycling mostly organic P in sewage sludge and manure [17] or in the form of valuable inorganic salts [18]. The potential for an efficiency increases of its usage is available all along the supply chain since currently P losses are estimated to amount of up to 95%, considering the chain from P rock to human consumption [19]. The global mass balance shows that up to 20% of the world's P production (about 3Mt P/year) is lost in anthropogenic waste [20]. P even being essential for all life on earth could cause in some cases of excessive use massive environmental pollution including eutrophication and long-term soil infertility [16]. The open cycle of phosphorus in nature also has serious ecological consequences. Excess phosphorus causes an imbalance of biosphere, leading to negative environmental effects. In Europe wastewater treatment (WWT) plays a key role to avoid eutrophication of surface waters and to recover phosphorus. The process merges all collected municipal and industrial wastewater streams for treatment and transfers them into sewage sludge and cleaned effluent. After digestion this P is discharged to wastewater via faeces and urine. Used detergents and other P containing products add another part to the flow [21]. While, previous efforts have been directed towards removing phosphorus from the wastewater discharged into surface water, nowadays efforts are directed to recovering P species [16]. Phosphorus resources follow a one-way pathway in nature, which is difficult to regenerate, leading to their increasing depletion. Although this problem is widely recognized, significant amounts of phosphorus resources are not being effectively recycled. As an example, China's municipal wastewater contains up to around 293 Mg/year of phosphorus, which equals approximately 5.5% of the chemical fertilizer phosphorus consumed in China [22]. Today's utilization of P is non-circular and dissipative. In the 1980s an investigation of Baccini and Brunner (1991) showed that the use of P is highly inefficient considering that only 10% of the applied P finds the consumer [21]. Meanwhile, a global mass balance shows that up to 20% of

the world's P production (about 3Mt P/year) is lost [23]. Shifting from linear to circular economy has consequences on how the sustainability of products are assessed [24]. This is the case for nutrients and organic matter which could be valorized as fertilizers and biogas (energy), amongst others [25]. Resource recovery from wastewater streams is increasingly seen as one option to help tackling challenges such as the resource efficiency of regions and countries and the low revenues from wastewater treatment [24]. The wastewater enters a recycling process named here "resource recovery processes" which consist of the wastewater treatment at the WWTP, the sludge processing and the processing of the recovered materials. Considering the new paradigm of waste-as-a-resource, several products related to the wastewater value chain have appeared; such as the food waste and sludge produced during the WWT. Thus, under the concept of sustainable development, domestic sewage or manure are no longer considered a waste but resources of different origin [4]. A general view of phosphorus consumption and pathways is given in Fig.1.

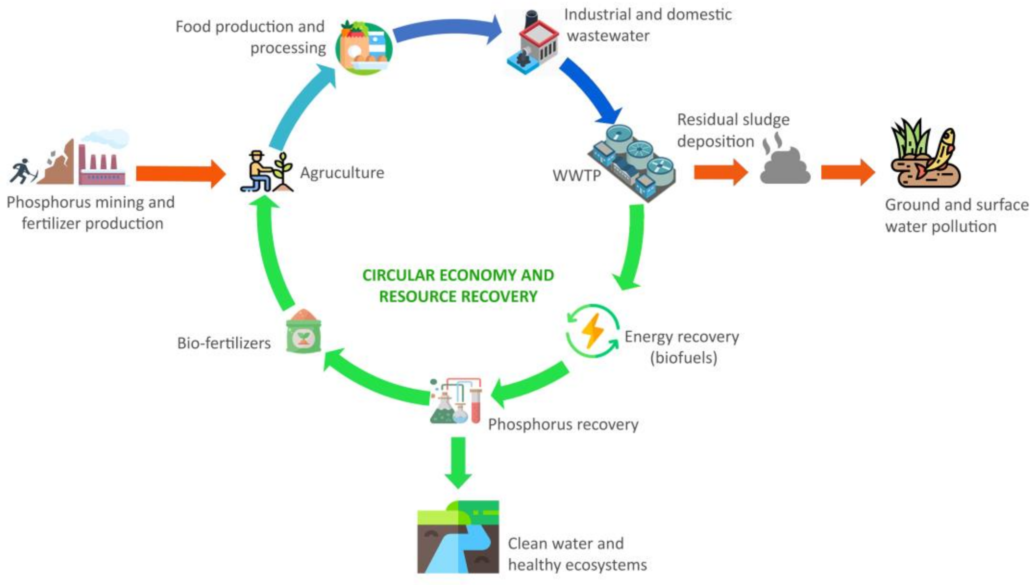

Fig. 1. Phosphorus consumption and pathways

Color code indicates the impact in terms of environmental health and sustainability:

Blue – neutral (currently applied practices); Red – Negative practices to be avoided or minimized; Green – processes with positive impact

3. Technologies for phosphorus recovery

As it was mentioned above, the shift into circular use of P passes through implementation of multidimensional innovations focused in creation of P recovery/recycling technologies. Despite apparent challenges, several technologies have been developed to recover P (as well as other nutrients and energy) from different waste streams [26]. Some of these technologies are now able to produce P-rich products for application in agriculture as fertilisers [26]. However, large-scale implementation of these technologies remains scattered, with only a few successful examples of full commercialization [27]. In Europe, a number of technologies for phosphorus recovery from wastewater are operating at either full or demonstration scale. Nevertheless, there are evidences that large-scale implementation of recovery is not profitable in the current market [28]. Even though interest in Europe-wide implementation of recovery exists [29]. Recovery technologies in the aqueous phase mainly focus on the sludge

water. In the solid phase, technologies either recover P from the dewatered sewage sludge or from mono-incinerated sewage sludge ashes [30]. The technologies differ in respect of technology choice, costs, efficiency and product purity. Chemical precipitation of slightly soluble salts is a common method for removing dissolved phosphorus from industrial wastewater: struvite (magnesium ammonium phosphate hexahydrate) or calcium phosphate [31]. Commercial large-scale struvite production plants which precipitate struvite from digested sludge liquors are operating in the USA, Canada, China and Belgium. However, in spite of the significant progress in struvite recovery technology, a huge room for struvite production optimization still exists. The recovered products can be utilized as fertilizer in agriculture or in specific industries. Consequently, closure of the anthropogenic P cycle through recovery and recycling of P from municipal wastewater and sludge, as well as from special industry wastewater, may help to avoid eutrophication, promote resource conservation and increase the value chain efficiency of this precious resource [31]. The main valuable renewable phosphorus sources are municipal waste, slaughterhouse waste, animal waste and other materials from food processing. It was estimated that one citizen generates around 2.5g of phosphorus per day in municipal waste [32]. Municipal and other wastes contain a considerable amount of phosphorus and there is a big potential to utilize it [16]. Most methods of phosphorus recovery from wastewater produce struvite [33]. This product can be directly used as fertilizer. This technology is especially useful when the wastewater also contains nitrogen [33]. However, in many cases, industrial wastewater treatment plants do not include nitrogen compounds if a biological wastewater treatment is not part of the whole process. In the case of plants producing wet phosphoric acid, a good solution is to precipitate hydroxyapatite as a process product and use it to partially replace the phosphoric raw material in the wet acid process [16]. Crystallization of struvite is a process for the simultaneous recovery of nitrogen and phosphorus [4]. Struvite is a white crystal that contains essential nutrients for plant growth and can be applied directly to the soil. It is considered an excellent fertilizer, because it minimizes the loss of nutrients due to its slow release rate and its low water solubility. The concentration of magnesium and the pH of the medium are limiting factors for crystal formation [34]. This technique of struvite crystallization can also be applied to the anaerobic digestion of sludge due to the high concentration of inorganic phosphorus and ammonia. The chemical precipitation of struvite can remove between 80 and 90% of the soluble phosphates and 20–30% of the soluble ammonia in the effluent. The main challenge in relation to struvite precipitation is the recovery of phosphorus from wastewater, if the phosphorus concentration is less than 50 mg/L and the concentration of suspended solids is above 2000 mg/L [4]. So, one possibility is to apply the struvite precipitation technique for secondary streams (such as generated in primary sludge thickening or after dewatering of the digested sludge) that have higher phosphorus concentrations. Phosphorus recovery is usually based on the chemical precipitation of struvite from the supernatant coming from anaerobic digestion of surplus sludge of wastewater treatment plants [35]. In addition, it is possible to recover phosphorus from the digested sludge before dewatering. For example, struvite can be precipitated in the sludge through acid leaching and subsequent chemical precipitation of phosphorus. As the concentrations of heavy metals present in the sludge or its ash are greater than the concentrations in the liquid phase, processes for decontamination need to be applied in such cases. However, the crystallization of struvite is considered a process which require optimization on recovery efficiency and application of alternative precipitation agents [36]. There are also struvite technologies that recover directly P from the solid phase by acidification. However, currently these technologies have not been implemented in large scale [30, 37].

3.1. Our experience on P Recovery

The *Water Treatment Lab at Burgas University* has initiated a P-recovery program with *Swiss National Science Foundation* (SNSF) within the Bulgarian- Swiss Bilateral Program on Innovative P-recovery by struvite precipitation

from waste municipal wastewater sludge liquor (DSL) and manure slurries. The main trends of studies were as follows:

- defining the optimal molar ratio $Mg:PO_4$ and pH;
- non-reagent pH elevation
- effectiveness of membrane separation of the organic matter;

Currently, the lab work is emphasised on bio-electrochemical dissolution of ferric phosphate in MWWTP sludge. Results related to molar ratio $Mg:PO_4$ and pH optimality are published in a study [38]. Here an example will be given for the experiment carried out with dewatered sludge liquor (DSL) taken from the MWWTP of Burgas (initial $PO4^{3-}$ concentration of 86.7mg/l). $MgCl_2$ was used as a precipitation agent and different pH and molar ratio $Mg:PO_4$ were applied, Table 1. Obviously, both the pH and mole ratio $Mg:PO_4$ are the crucial factors of precipitation efficiency. However, highest effect is observed at pH over 9. At pH >11.0 the struvite obtained decreased due to the formation of $Mg(OH)_2$ and the evolution of free NH_3 [39], thus reducing the Mg^{2+} and NH_4^{+} ions available for struvite formation. Experiments carried out by Saidou et al. [40] showed that for the initial solution pH of 10, another phosphate mineral, namely $Mg_3(PO4)_2$. $2H_2O$ starts precipitating. Such formation is the results of the dominant species of phosphate ions in the form of PO_4^{-3} in the solution. In fact, varying the pH levels results in different species of phosphate ions: PO_4^{3-}, HPO_4^{2-}, $H_2PO_4^{-}$ [39]. The precipitation of struvite requires equal molar ratios of its components: Mg, N, and P [41]. Different molar ratios have shown to influence several characteristics. It can be argued that as more Mg is available for the reaction, the more crystal units are generated, hence larger crystals may form. As reported by Merino-Jimenez et al. [43], several studies have applied the ratios of PO_4 and Mg around 1:1.2 for optimum struvite yield [44]. The later result confirms our results which show better precipitation rate at $Mg:PO_4$ >1.

Table 1. Series of experiments using supernatants produced after centrifugation of sludge from WWTP-Burgas.

Molar ratio $Mg:PO_4^{3-}$	1:1	2:1	3:1	1:1	2:1	3:1	1:1	2:1	3:1
pH	8	8	8	9	9	9	10	10	10
P Removal efficiency, %	45.6	44.7	44.9	76.1	76.3	78.4	79.9	89.9	89.2

In order to increase the yield of struvite through its precipitation in MWWT sludge dewatering liquor it is important to control the Organic Matter (OM) content. There are evidences that the particulate OM is influences negatively the struvite crystallization kinetics and the characteristics of precipitates. In addition, the OM causes lower Mg salts dissolution by steric effects [44]. The potential explanation is that the OM prevent the dissociation of $Mg(OH)_2$ into Mg^{2+} and OH^{-}. As it was shown above one of the main factors for struvite formation is the solution pH. It can be adjusted by non-reagent carbon (CO_2) dioxide stripping by aeration. The intensity of the mass transfer between the air and the supernatant of dewatering sludge obtained from wastewater treatment plant is characterized by the volumetric liquid-side mass transfer coefficient, which can be estimated theoretically. It is found [45] that the rate of pH increase depends strongly on the sparging area of the air distribution system while the air flow rate does not influence considerably the Dissolved Oxygen level which governs the CO_2 stripping process. The theoretical calculated values of the volumetric mass transfer coefficient have been compared with those obtained experimentally. Based on the data

obtained, relationships of pH/*kLa* (mass transfer coefficient) were developed. These correlations serve as a tool for prediction of pH during the struvite precipitation process. The pH dependence of air rate is given in Fig.2.

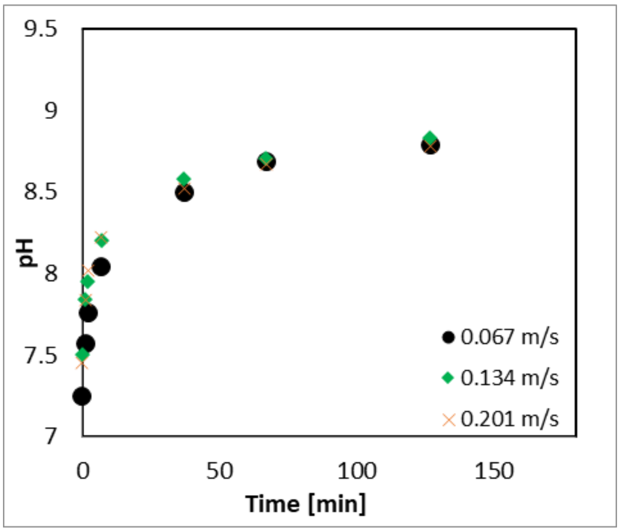

Figure 2. pH elevation by air stripping through applying different volumetric air rates

The results show evidently that within the volumetric air rates applied pH increases from 7.5 to 8.4 were achieved by air stripping in less than 20 minutes. Actually, pH value around 8.5 is enough high for struvite precipitation even in several studies the optimal pH is shown to be in the range of 9 to 9.5. The targeted pH of 9 was achieved in a moderate mode. However, the slow change of pH after the 20th minute of aeration is an advantage of the CO_2 stripping process because it restricts the rapid increase of solution saturation. As such conditions the struvite crystallization process predominates [45]. Within our experiments the techniques of microfiltration (MF) and nano-filtration (NF) were tested as possible options to separate the particulate organic matter from soluble nutrients after sludge dewatering by centrifugation. By further treatment of the filtrate it is aimed to valorised nitrogen and phosphorus [46]. Microfiltration with flat sheet membranes (membrane pore size of 6 µm) was applied at dead-end mode and 2 bar pressure. The results obtained show a fast decline of permeability rate (l/l.m2), by 60% within the first hour of filtration and 93% at the elapsed hour "four". In case of swine wastewater filtration, the rate decreased by 98% after 4 hours. Similar short operation periods are reported for dead-end microfiltration [47].

Table 2. Results obtained through the nanofiltration process

UF membrane (pore size, nm)	Initial COD in the sludge, mgO_2/l	Residual COD in permeate, mgO_2/l	COD removal level, %	Concentration of PO_4 in permeate, mg/l
50	78100	414	99.47	118
200	78100	642	99.18	195

The initial acceptable performance is followed by filter blocking which limits membrane utilization for practical application**.** A lab-scale UF cross-flow unit for organic matter/nutrients separation was in use. The unit operated at cross-flow mode was equipped with ceramic tubular membranes of CMF19033 type (made of 99 % α-Al_2O_3/ZrO_2). UF tubes with of pore sizes of 50 nm and 200 nm were in use. The filtration runs were carried at 4 bars. The filtration system was fed with dewatered sludge liquor characterized by TSS of 26 g/l, o-PO4 $^{3-}$ of 250 mg/l and COD of 78.1

g/l. The results obtained are given in Table 2 and reveal that the highest effect of organic matter retention was achieved with the lower pore size membrane, even the difference is negligible. However, the 200 nm membrane is more appropriate to be accepted as it keeps much higher level of phosphorus in permeate. In case of application of membrane module with pore size of 200 nm, the chemical analysis of the permeate show a residual phosphate concentration of 195 mg/L, while COD is 642 mgO_2/L and TSS - 0.15 g/L and ammonia concentration - 224 mg/L. The UF phosphorus reduction (200 nm pore membrane) is 22 % which makes the separation successful. In further experiments with the permeate obtained, the extent of P removal through struvite precipitation was around 90%. This result coincides with Struvite crystallization rate observed in the specialised literature, which corresponds to 80-90% of $PO4^{3-}$ reduction [48,49, 50].

4. Discussion

Phosphorus is an essential element in sustaining modern day farming practices and is expected to deplete within the coming 50-100 years [51]. However, phosphorus utilisation efficiencies in most countries are below 20%, making the implementation of suitable phosphorus recovering technologies urgent and necessary. In spite of intensive research and development, there are only a few commercial recovery facilities being implemented [52]. The experience in struvite precipitation shows that this product has characteristic allowing the recovered phosphorus to be applied to soil at rates greatly exceeding those of conventional fertilizers without damaging plant roots [53]. In addition, struvite crystallization process has the advantage of CO_2 abatement of about 100 kg/t less than traditional phosphate rock extraction [54], with a cost of CO_2 emissions is between 20 and 120 US D a tonne, which generates an additional environmental profit. Although phosphorus is not nowadays figured scarce in the short and midterm, we have to manage it carefully and take responsible decisions for sustainable ways of use of this limited resource. In this sense the ***innovation*** should be taken as the only key to move toward sustainable P management [55]. Accepting that P recovery from waste is one of the most promising innovation of the modern technology, the advantages of this option should be underlined. Its application in the narrow aspect of application in municipal wastewater treatment is featured by

- Reduction of P load to the headwork of wastewater treatment plants (WWTP); If the sludge liquor returns to the headworks as is most often the case, it increases the P loading in the influent stream of the plant by up to 40% [56].
- Improved sludge management in advanced WWTP in sense of decreasing of sludge volume and improvement of its dewatering characteristics.
- Eliminate the danger of potential struvite formation in MWWTP equipment caused by the high phosphate and ammonia levels, levels typical for anaerobic digester liquors (the anaerobic liquor have been reported to reach concentrations of 100 mg/l).

Regarding the nutrition capacity, struvite is a highly effective fertilizer across a broad range of soil pH levels [57]. Compared with phosphorus fertilizer made of phosphate rocks, struvite recovered from wastewater has lower concentrations of *heavy metals* and radioactive elements. In addition, the view of experts [58] suggest that struvite is considered a potentially suitable fertilizer for *organic farming* since chemical inputs and processing during P recovery and fertilizer production are low. Being a slow-release fertiliser struvite is superior to the regular manufactured mineral fertilizers because it does not 'burn' roots as well [59-61]. Struvite technology possesses also the perspective to be applied more intensively for manure treatment. The manure as an waste media which contains much higher P/N levels than the sludge liquor originates from MWWTP. This attracts the attention in attempts to find a suitable phosphorus (mainly) waste source. However, the contemporary technology should alleviate the problems created with

the high organic matter content of manure and the subsequent need for efficient separation of the fine suspended/colloidal OM from the soluble nutrients.

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