

Struvite Precipitation Technologies

Subjects: Others

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The abatement of nutrient compounds from aqueous waste and wastewater is currently a priority issue. Indeed, the uncontrolled discharge of high levels of nutrients into water bodies causes serious deteriorations of environmental quality. On the other hand, the increasing request of nutrient compounds for agronomic utilizations makes it strictly necessary to identify technologies able to recover the nutrients from wastewater streams so as to avoid the consumption of natural resources. In this regard, the removal and recovery of nitrogen and phosphorus from aqueous waste and wastewater as struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) represents an attractive approach. Indeed, through the struvite precipitation it is possible to effectively remove the ammonium and phosphate content of many types of wastewater and to produce a solid compound, with only a trace of impurities. This precipitate, due to its chemical characteristics, represents a valuable multi-nutrients slow release fertilizer for vegetables and plants growth. For these reasons, the struvite precipitation technology constantly progresses on several aspects of the process.

Keywords: ammonium ; magnesium ; MAP ; nutrients recovery ; phosphorus ; struvite

1. Introduction

The definition of proper processes for the management and the treatment of waste and wastewater is a crucial environmental and social topic. The uncontrolled discharge can induce serious deteriorations of soil and water bodies quality. In particular, the release in the environment of aqueous waste with high amounts of nutrients (nitrogen and phosphorus compounds) is of great concern ^{[1][2][3][4]}. In fact, this practice induces eutrophication phenomena in superficial water bodies and, consequently, the exhaustion of O_2 content ^[5]. As a consequence of eutrophication phenomena, episodes of harmful algal blooms (HAB) could occur. HAB can release harmful toxins which pose a notable risk for human health and environmental ecosystems ^[6]. Additional drawbacks take place as some nitrogen compounds (NH_3 , NO_2^- , NO_3^-) are harmful to aquatic life or may cause disease if drinking water is polluted with these elements ^[7]. The accumulation of nutrient compounds in aquatic environments is mostly attributable to the use in agricultural activities of chemical fertilizers, animal manures and digestates, as well as to the discharge of urban and industrial wastewater ^{[4][8][9][10][11][12][13][14][15]}.

Numerous biological and physical-chemical treatments for the abatement of nutrients have been developed. The nitrogen compounds can be removed through typical biological nitrification–denitrification processes ^{[5][15][16]}. These processes are widely used and effective for NH_4^+ removal in municipal treatment plants, but they are not very suitable for treating high concentrated wastewaters ^[17]. Indeed, a high ammonium content has toxic effects on microorganisms, which may cause a significant reduction in the process efficiency ^[1]. Shortcut nitrification/denitrification and anaerobic ammonium oxidation, showed potential to overcome the drawbacks above mentioned and they could be applied in the treatment of high polluting matrix such as leachates, digestates, etc. Nevertheless, these techniques are still under development and they are quite difficult to control and manage ^[17].

In the context of chemical-physical treatments, ammonium stripping is one of most widely employed treatments for the abatement of NH_4^+ from highly polluted wastewater. In this process, typically, the NH_3 , transferred from the wastewater stream to the air flow, is absorbed into acid solutions and recovered as ammoniacal salts. In spite of its benefits, this technology has some limitations, such as the need of pH adjustment to values close to 11–12 and the requirement of high air flowrates ^[18].

Breakpoint chlorination can also be applied to oxidize ammonium to nitrogen gas. However, because the process has a number of disadvantages, including the buildup of HCl and of total dissolved solids, and the formation of unwanted chloro-organic compounds, ammonia oxidation is seldom used today ^[18].

The abatement of phosphorus from municipal wastewater can be obtained in biological treatment plants which promote the growth of P-accumulating microorganisms. Many different processes have been developed (photoredox A/O, A²/O, Bardenpho, etc.) that assure good efficiencies [18]. A limitation of many of these processes is the occurrence of a decline in phosphorus removal when the nitrogen abatement is contextually applied [18]. In wastewater treatment plants the P abatement and recovery is also conducted through the precipitation of phosphates with the addition of coagulants which generally consist of inorganic salts of multivalent metal ions, such as calcium, aluminum, and iron. These processes are widely applied due to the easy management and simplicity of the required equipment. Moreover, they can be exploited also in the treatment of concentrated aqueous waste and wastewater. On the other hand, the use of coagulants generates high amounts of voluminous sludge which incorporates the organic solids resulting in a heterogeneous matrix which is difficult to manage.

Additional techniques, such as adsorption, ion exchange, reverse osmosis, etc., could be potentially applicable to remove nutrient compounds [3][18]. However, these techniques are not selective and generate concentrated streams, of which disposal is very expensive.

Among the many alternatives, a solution based on nutrients recovery and reuse is very advantageous as it promotes the development of sustainable technologies. In effect, the definition of a virtuous cycle of nutrients to reduce the use of natural resources and, at the same time, avoid pollution phenomena is, currently, a key challenge. The recovery of nutrients is a strict necessity especially in the case of phosphorus. In fact, nowadays phosphorus is mainly obtained by extractive activity [19][20]. The extractive peak of phosphatic rocks will be reached in the next decades and the phosphorus reserves can decrease drastically in the next century [20]. When phosphate rocks drop, extraction and marketing prices will also rise, and industry will be forced to seek alternative sources due to higher demand of phosphorus in the agricultural world. Therefore, the attention of the international scientific community is directed towards the search for sustainable sources of phosphorus and more generally of nutrients. In this regard, the European Commission recently revised the EU Fertilizer Regulation ((EC) 2003/2003) and published the new EU Regulation ((EU) 2019/1009), which includes the fertilizing compounds based on secondary raw materials [10].

Many raw materials recoverable from the treatment of aqueous waste and wastewater could potentially become component materials in the new Regulation ((EU) 2019/1009) [10]. In this regard, the recovery of ammonium and phosphates from wastewater as struvite has gained great interest in recent years. Struvite is a white orthorhombic crystalline compound, which is composed of Mg²⁺, NH₄⁺, and PO₄³⁻ (MgNH₄PO₄·6H₂O) in equal molar amounts [21][22]. Struvite precipitation takes place when the combination of ammonium, magnesium, and phosphates concentrations overcomes the solubility product under an alkaline environment [23]. In general, struvite precipitation can be aimed to remove and recover the amount of NH₄⁺ or PO₄³⁻ from wastewater [8][24][25][26]. The recovery of the phosphates content alone is much more sustainable because lower amounts of reagents are necessary. In fact, at most, the chemicals for magnesium supplying and pH setting are required. In the case of nitrogen removal, often, due to the limited contents of Mg²⁺ and PO₄³⁻ with respect to the NH₄⁺ concentration, higher dosages of the reagents are needed for the treatment of high concentrated wastewater [1].

Anyway, struvite is widely recovered for nutrients recycling from wastewater because it can be produced a relatively pure precipitate with a great amount of both phosphorus and nitrogen, with only trace amounts of impurities [10]. Indeed, unlike other precipitation processes that exploit coagulant agents, the struvite is formed as a mineral salt that incorporates only low quantities of solids and that can be easily recovered by sedimentation [27,28]. Therefore, the formation of voluminous flocculant sludge, containing great quantities of unwanted compounds is avoided. Furthermore, because of its chemical composition, struvite has a demonstrated value as a fertilizer (pure struvite is included in the Regulation EC No 1907/2006 (REACH) as a commercial fertilizer) [27][28][29][30][31][32][33]. For these reasons, at present, struvite is the most common recovered compound in pilot and operational facilities in Europe [10]. It was estimated that approximately 15,000 tons are produced in Europe annually [10]. The existing facilities mainly treat urban wastewater to produce struvite, but industrial wastewater, manure, and livestock slurries are also exploited as input aqueous waste [10]. Furthermore, significant struvite quantities are produced outside Europe (USA, Japan, China) [10].

Despite several applications in industrial facilities, the struvite precipitation process is under continuous development. Indeed, a lot of research has been recently carried out to solve some crucial aspects and to improve the process globally.

2. Full-Scale Struvite Recovery Technologies

In addition to the numberless applications at laboratory and pilot scale plants, currently, due to its profitable aspects, the struvite recovery processes are applied at industrial scale for the treatment of municipal and industrial wastewater and agro-industrial waste [19]. The most used industrial-scale technologies include:

- OSTARA (Pearl®). Ostara technology uses a FBR for struvite crystals recovery consisting of three zones with an increasing diameter from the bottom to the top. In the lower area, there is reagents feeding and recovery of struvite crystals; in the central one the nucleation and growth of the crystals take place; while in the upper part, the discharge of the treated wastewater occurs. The effluent is recirculated with a flow rate of about 20 times the inlet flow [34]. $MgCl_2$ is added to guarantee the molar ratio Mg:N:P equal to 1:1:1, while NaOH is added as an alkaline reagent [19]. This technology has been developed since 2005 by the University of British Columbia. Currently, 22 plants that use this technology are installed all over the world, most of which are located in the USA and Canada. Ostara technology allows up to 22% of total phosphorus to be recovered in an urban sidestream wastewater treatment plant, and up to 95% of phosphorus and 15% of ammonia nitrogen in the treatment of digestion supernatants [35]. Furthermore, the process allows daily productions of about 500 kg/d of struvite particles with sizes between 1.5 and 4.5 mm [20].
- SEABORNE. The Seaborne process was developed in 2006 in Germany and it involves nutrients recovery from digested sludge pretreated with sulfuric acid [35]. The technology consists of a first acidification phase with H_2SO_4 to extract nutrients and metals. The remaining solid phase is incinerated while the liquid phase undergoes a process for the recovery of the heavy metals by gas-liquid precipitation using H_2S -rich biogas [35]. Subsequently, nutrients recovery as struvite takes place in a completely mixed reactor after the addition of $Mg(OH)_2$, as a magnesium source, and NaOH to reach the pH 9. Finally, the residual dissolved ammonium is recovered through a scrubber in the form of ammonium sulfate [19][20].
- PHOSPAQ™. This process, developed in Holland, aims at the removal both of phosphorus and ammonium present in the wastewater. The process takes place in a completely mixed aerated reactor to obtain the simultaneous degradation of COD and nutrients recovery in the form of struvite. Aeration permits the biological COD oxidation and, at the same time, the CO_2 stripping which favors the establishment of pH values around to 8.2, suitable for the struvite precipitation [19]. MgO is dosed as a source of Mg^{2+} to reach the stoichiometric N:Mg:P molar ratios. With this technology it is possible to produce MAP particles with dimensions of about 0.7mm [19][20]. Phosphorus removals between 75% and 81% were obtained in an industrial-scale application conducted on potato processing wastewater.
- ANPHOS®. This technology for phosphorus recovery was developed in the Netherlands and operates in batch conditions. In particular, in a first aerated reactor the waste alkalization takes place by CO_2 stripping, while in a second reactor, $Mg(OH)_2$ is introduced as a source of Mg^{2+} to promote the MAP formation. This process can recover 80–90% of the phosphorus contained in the influent. Once dried, the produced struvite can be directly used as a fertilizer [20]. With this treatment, a reduction in the influent COD also occurs, which leads to a lower oxygen consumption in the subsequent treatments [20].
- PHOSNIX. The process involves the phosphorus recovery as struvite from supernatants of digested sludge in a fluidized bed reactor [36]. This process was developed by the Japanese company Unitika Ltd. Environmental and Engineering Division. $Mg(OH)_2$ is dosed as a source of magnesium, until the molar ratio of Mg:P of 1:1 is reached to allow struvite crystals nucleation and growth. pH values between 8.2 and 8.8 are guaranteed by an aeration system and the addition of NaOH, which also promote the ammonium stripping. The process allows recovering struvite crystals directly usable as fertilizers between 0.5–1 mm in size [36].
- NuReSYS. The NuReSYS (Nutrients Recovery System) process was developed in Belgium for struvite recovery from digested sludges and dewatering effluents. This process allows the continuous recovery of the phosphorus using a completely mixed reactor [36]. The pH suitable for struvite crystallization is guaranteed by an aeration system which induces the CO_2 stripping. The process can operate in two different modes. Specifically, it can be applied to favor the spontaneous precipitation of struvite, without adding magnesium, to avoid the scale formation on the sludge management equipment. The other configuration, instead, exploits the introduction of $Mg(Cl)_2$, as a source of magnesium, to allow better efficiency in phosphorus removal. Phosphorus abatement about 96% and a production of struvite particles of variable sizes between 2 and 6 mm, were obtained with NuReSYS process [37].
- AirPrex®. AirPrex technology was developed in Germany to solve the struvite incrustation problems downstream of the anaerobic digestion processes [38]. This technology uses two reactors for struvite precipitation and recovery from digestates before dehydration treatment. In the first reactor, the nucleation and growth of struvite crystals take place. This unit is aerated to promote the pH increase around 8 and to keep the struvite crystals in agitation. Two vertical partitions further promote an effective circulation. The second conical bottom reactor allows the sedimentation of struvite crystals and the discharge of the purified effluent [20].
- MULTIFORM™. Multiform technology was developed in the United States for nutrients recovery from agro-industrial wastewater. The plant unit consists of a cone-shaped fluidized bed reactor that allows to remove about 80% of the phosphorus from wastewater with a struvite crystals retention time equal to three days [39]. Crystallization is favored by

adding $\text{Mg}(\text{Cl})_2$, as a source of magnesium, and NaOH as an alkaline reagent. Although the struvite crystals are produced quickly, the crystals obtained have a low degree of purity [39].

3. Use of Struvite as a Fertilizer

Struvite has some characteristics very favorable for the use in agronomic applications. First of all, struvite contains essential nutrients such as nitrogen and phosphorus for plant growth [40]. Furthermore, its low solubility in environmental conditions induces a slow release of ammonium and phosphorus in the soil, which allows optimal plant growth over the time, avoiding potentially harmful overdose phenomena [41]. Finally, the struvite precipitation from waste and wastewater recovers nutrients that would be otherwise dispersed in the environment, saving environmental and economic damages [42]. Zhang et al. [41] calculated that for every 100 m^3 of treated livestock wastewater, up to 1 kg of struvite can be recovered, allowing to avoid the dispersion of nutrients in surface water bodies.

Anyhow, the potential reuse in agronomic practices should satisfy specific legal requirements. Clearly, the recovered compounds must not contain hazardous elements able to produce environmental impacts or human health damage. The EU Fertilizing Products Regulation ((EU) 2019/1009) [43], in the case of inorganic macronutrient fertilizers, establishes threshold values for the following elements: Cd ($60 \text{ mg/kg}_{\text{P}_2\text{O}_5}$), Cr^{6+} ($2 \text{ mg/kg}_{\text{dry matter}}$), Hg ($1 \text{ mg/kg}_{\text{dry matter}}$), Ni ($100 \text{ mg/kg}_{\text{dry matter}}$), Pb ($120 \text{ mg/kg}_{\text{dry matter}}$), As ($40 \text{ mg/kg}_{\text{dry matter}}$), $\text{C}_2\text{H}_5\text{N}_3\text{O}_2$ ($12 \text{ mg/kg}_{\text{dry matter}}$), ClO_4^- ($50 \text{ mg/kg}_{\text{dry matter}}$), Cu ($600 \text{ mg/kg}_{\text{dry matter}}$) and ($1500 \text{ mg/kg}_{\text{dry matter}}$). In addition to these limits, a fertilizer containing more than 1% by mass of organic carbon shall meet requirements for pathogens [43]. Similarly, the legal regulations in other countries fix limits for heavy metals and organic micropollutants [31][44]. Therefore, it is essential to recover a precipitate characterized by limited amounts of hazardous elements. In this regard, struvite has been shown to be characterized by a low content of heavy metals and dangerous micropollutants, such as PCBs [31][45].

Several studies were carried out to evaluate the effects on plant growth using struvite recovered from wastewater as a fertilizer [32][46][47][45]. Struvite was tested as a slow-release fertilizer in pot plants, vegetable crops, ornamental plants, turf grass, and field crops [48]. El-Diwani et al. [49] conducted a comparative study on broad bean growth rate using a commercial fertilizer and struvite recovered from the treatment of industrial wastewater. In particular, the study compared the broad bean growth in absence of fertilizers with struvite and using a mixture of pure fertilizing compounds. The struvite was dosed at a rate of 2%, whereas, in the sample with pure compounds, nitrogen, phosphorus, and potassium were applied at a rate of 20, 50, and 40 $\text{mg/Kg}_{\text{soil}}$ as ammonium nitrate, superphosphate, and potassium sulphate, respectively. The results of plant growth tests showed that the application of struvite increased the fresh and dry weight of broad bean plants at different stages of growth.

Li and Zhao [48], to assess the fertilizing power of the struvite, recovered from landfill leachate, executed a set of pot tests using four species of vegetables including Chinese flowering cabbage, Chinese chard, water spinach, and water convulvulus. Each plant was growing without added fertilizer, with struvite supplementation, and with the feeding of a model fertilizer. The germination and growth of the vegetables in the pots with struvite showed notably higher rates compared to those in control tests [48]. The experiments also proved that, due to its poor solubility in water, the struvite overdosing did not cause any problems. Furthermore, the authors observed that the MAP utilization did not result in more heavy metals in the vegetables than those from control and model fertilizer [48]. Yetilmezsoy and Zengin [50] also studied the potential use of struvite, recovered from UASB treated poultry manure wastewater, through a series of tests with fast-growing plants such as portulaca (*Portulaca oleracea*), garden cress (*Lepidium sativum*), and grass (*Lolium perenne*). In particular, a comparison was made using garden soil as control; garden soil with addition of struvite; sand with addition of KCl as control; sand with addition of KCl and MAP precipitate.

The experiments showed that the plants that were treated with struvite grew much faster than the garden soils and the samples with only KCl. Moreover, the application of struvite notably increased both fresh and dry weights of the plants up to 257% and 402%, respectively [50].

Liu et al. [51] conducted a study using MAP recovered from swine waste as a slow-release fertilizer for corn growth. In this study, plant growth was evaluated on an unfertilized soil (control), on a soil fertilized with struvite, and on a soil fertilized with a generic chemical fertilizer, consisting of a superphosphate (20% of P_2O_5) and urea (46% of N) [51]. The results showed that struvite can be successfully applied to the cultivation of corn in pots as the plants' heights and circumferences, as well as the nutritional components, were similar in plants fertilized with MAP and with generic chemical fertilizers [51].

Ryu et al. [45] compared the fertilizing power of struvite recovered from a semiconductor wastewater treatment plant with those of complex commercial fertilizers and organic compost for Chinese cabbage cultivation. The tests showed different growth rates in relation to the type of used fertilizer. In particular, the tests carried out with struvite showed the second-best growth rate, second only to the samples treated with the complex fertilizer [45]. The best performances of the complex fertilizer have been identified in the greater supply of potassium (K). The lowest accumulation of Cu and no detection of Cd, As, Pb, and Ni was observed in the struvite-treated samples. Overall, a dosage of $1.6 \text{ g}_{\text{struvite}}/\text{kg}_{\text{soil}}$ was identified to effectively grow Chinese cabbage [45].

Uysal et al. [32] conducted a study on tomato and corn plant growth using struvite recovered from the anaerobic effluent of the baking yeast industry. Four different sets of experiments were performed to evaluate struvite effect on crops: a set without the use of any fertilizer, a set with NH_4NO_3 , a set with the addition of fertilizer based on NH_4NO_3 and KH_2PO_4 , and finally, a set in which the recovered struvite was added with four different dosages (2.85, 5.71, 8.58, $11.42 \text{ g}_{\text{struvite}}/\text{kg}_{\text{soil}}$) [32]. The results showed that with the minimum dosage of struvite ($2.85 \text{ g}_{\text{struvite}}/\text{kg}_{\text{soil}}$) the same quantitative results were obtained as commercial fertilizer, while the best result in terms of dosage was equal to a quantity of struvite of $5.71 \text{ g}_{\text{struvite}}/\text{kg}_{\text{soil}}$ [32].

Siciliano [46] carried out agronomic tests to analyze the fertilizing potential of struvite precipitate recovered from the treatment of methanogenic landfill leachate. The fertilizing effects of MAP in cultivating *Spinacia oleracea* were compared with those of vegetable soil and of a commercial fertilizer [46]. The growth of vegetables in the pots with MAP was notably higher than those in the pots with only garden soil and in the pots with the fertilizer. Moreover, the struvite utilization as fertilizer did not result in an increase of metals absorption by vegetables [46].

These studies confirmed the effectiveness of MAP recovered from aqueous waste and wastewater as a slow release fertilizer. In effect, as previously discussed, struvite is produced at industrial scale and, among the different processes, the struvite products Pearl and NuReSYS are certified as fertilizers in the United States, United Kingdom, and Belgium, respectively. In addition, the struvite obtained by the Seaborne process is used locally.

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