Research article

Recycling of nutrients from landfill leachate: A case study

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HIGHLIGHTS

- Landfill leachate can be used as a source of nutrients for the growth of maize by precipitation of struvite.
- A field trial in real scale was performed.
- A marginal higher maize yield was achieved in two of the sites (6.36% and 2.16%) when compared to the commercial fertilizer.
- Struvite did not cause presence of pathogens or heavy metals in the crops.
- It offers an alternative to conventional leachate treatment options, aligned with the principles of the circular economy.

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ABSTRACT

The continuous increase in the consumption of natural resources requires different solutions directed to the recovery and recycling of different materials and products, including the nutrients used as fertilizers for food production. In this context, this research assessed the feasibility of using landfill leachate as a source of nutrients for the growth of maize. Leachate was treated to precipitate struvite, a rich magnesium, phosphate, and ammonium mineral that can be applied directly as fertilizer. It was used for the growth of maize, which was sowed in three different parcels. A commercial DAP + urea mixture was used to compare, and non-fertilized parcels were used as controls. Struvite was successfully obtained and applied in the fields. A marginal higher maize yield was achieved in two sites when using struvite (6.36% and 2.16%) compared to the commercial fertilizer, even if it was applied in a lower dose to weather conditions. An increase in N and Mg in soil could be observed, which allowed for the assimilation of nutrients in the plants. Concerning safety, the use of struvite did not produce the transfer of heavy metals or pathogens to the soil or plants. This research shows a promising way of dealing with leachate, which could be attractive in countries where organic waste is buried in landfills.

1. Introduction

The possibility of nutrient recovery and recycling is considered one of the key elements to move towards the circular economy. It would reduce the depletion of natural resources, the impact of their extraction and manufacture (Krishnamoorthy et al., 2021; Robles et al., 2020). However, when nutrients such as nitrogen and phosphorous are not recovered, they can become an environmental problem, affecting the quality of water bodies and land. This pollution could happen due to fertilizers runoff (Cui et al., 2020; Wang et al., 2019), discharges from livestock (Li

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et al., 2020; Rothwell et al., 2020), and the production of organic solid waste (Luo et al., 2020a, b; Wainaina et al., 2020).

In many developing countries, organic waste is frequently disposed of in landfills and dumpsites (World Bank Group, 2018), producing global warming gases and leachates. Landfill leachate can be heavily polluted and must be treated by biological and physical-chemical processes, frequently combined, to reduce its impact on the environment (Luo et al., 2020a, b). Treatment can be challenging due to the presence of both organic and inorganic pollutants, including ammonia nitrogen. Treatment processes that can remove nitrogen include heating (Schwarzwalder Sprowieri et al., 2020), filtration, oxidation (Wang et al., 2020), adsorption (Halin et al., 2010), and osmosis (Iskander et al., 2018), among other techniques. One of the alternatives to removing nutrients in nitrogen-enriched wastewater and landfill leachate is through precipitation of struvite, which is obtained in the presence of magnesium and phosphorus (Tansel et al., 2018). The production of struvite has also been successful to remove phosphate on industrial wastewater (Nandra et al., 2021).

Struvite is a hydrated compound with equimolar concentrations of Mg, ammonium, and phosphate (MgNH4PO4·6H2O). It is characterized by orthorhombic crystals, which can be pyramidal or tubular, among other shapes, with white-yellow color (Anthony et al., 2001). It has a molecular weight of 245.43 g/mol, it has low solubility in water in neutral and basic pH (<5%), but is easily soluble in acidic media (Chirumuley, 1994; Gu et al., 2021). It can be used as a slow-release fertilizer due to its low content of heavy metals (Hu et al., 2020; Sánchez et al., 2011). It has been shown that the use of struvite as a multi-element fertilizer prevents water pollution due to its low solubility, preventing washing by rain and irrigation (Li et al., 2019; Mikula et al., 2020). It can help reduce the extraction of resources due to phosphorus, which faces the risk of depletion (Nesme et al., 2018). It also diminishes the high energy consumption of fertilizers containing nitrogen (Land and Water Division, 2002), and can be used to supply phosphorous in hydroponic production (Arcas-Pilz et al., 2021). The possibility of recycling nutrients is desirable for countries like Mexico, where agriculture is a relevant economic activity; in 2019 Mexico was the 7th producer of maize worldwide, based mainly on small farmers, which account for 60% of the production (SADR, 2019).

In this context, this research evaluates the feasibility of recycling nutrients extracted through the precipitation of struvite from landfill leachate and its use as a fertilizer in the growth of white maize in a full-scale field test. As urban solid waste in Mexico contains 46.62% of organics, and is mainly buried in landfills without segregation (SEARNA, 2020), the production of leachate with high nutrients content can be expected. For this research the leachate was obtained from the treatment plant of the landfill “Los Laureles.” Struvite was applied in plots located in the agricultural area close to the landfill in the state of Jalisco, which traditionally has been the second producer of maize in the country, with 14% of the total production (ASCDMA, 2018). This study aimed to assess the comparative yield of maize produced in struvite-fertilized plots and evaluate possible risks due to the dissemination of pathogens and plagues. Regarding the addition of fertilizers, the basic idea was to provide the required dosage of nitrogen. The Mexican national authority for agriculture, SAGARPA, recommends a dose of 240 kg of N for ha to produce eight tons of maize (SAGARPA & INIFAP, 2015). A mass balance, shown in Table 1, was performed to define the required dosage of fertilizers, based on their composition (De Vries et al., 2017; Lezip et al., 2015).

Local farmers apply 250 kg/ha of DAP (45 kg N/ha) and 500 kg/ha of urea (200 kg N/ha), i.e., a total N load of 245 kg/ha. While DAP is applied during sowing, 200 kg of urea are added between days 24–30 after sowing, and 300 kg at tasselling. The mass balance resulted in a struvite requirement of 8,166.92 kg/ha, applied in different stages. However, only the first dosage of 2,144.16 kg/ha was applied due to the flooding of adjacent lands, limiting access to the plots during the test. Due to this situation, only 26.25% of the theoretical requirement of struvite was applied, as shown in the supplementary material.

2.2.2. Sowing, addition of fertilizers and pesticides

The sowing was performed according to local practices regarding the use of machinery, irrigation, and the application of pesticides to prevent plagues. Regarding the addition of fertilizers, the basic idea was to provide the required dosage of nitrogen. The Mexican national authority for agriculture, SAGARPA, recommends a dose of 240 kg of N for ha to produce eight tons of maize (SAGARPA & INIFAP, 2015). A mass balance, shown in Table 1, was performed to define the required dosage of fertilizers, based on their composition (De Vries et al., 2017; Lezip et al., 2015).

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Irrigation was performed according to local practices (seasonal rainwater irrigation), guaranteeing similar conditions for plots located in each area. The most common plagues for maize in the area are Spodoptera frugiperda, Helicoverpa zeamays, and Atta Mexicana. According to local practices, the pesticides Flash Ultra, Calibre 90, Anaclor and Glifosato were added to prevent their attack.

2.2.3. Assessment of the process

The growth of maize was monitored by inspection of the size and conditions of the plants. The harvest was done manually to allow the separation of the product in the different experimental treatments. The assessment of the process for each combination of soil and fertilizer had two main objectives: to assess the efficiency of struvite as fertilizer and verify that it does not transfer heavy metals, pathogens, or other undesirable characteristics to the soil or the grain. For this purpose, the yield of the field (for grain and maize crop biomass) was calculated, and a linear regression analysis was performed to identify possible relationships between Mg concentration and yield of maize. The characterization of soil and grain was performed according to the methods listed in the Supplementary material.

The samples of soil were sundried, mixed, and sieved (<2 mm). They were digested following the method EPA 3051A (EPA, 2007), with an acid solution containing 25% of HCl and 75% of HNO3 (vol/vol) in a microwave (CEM, MARSX press) for 25 min. Grain samples were also dried, mixed, and sieved, and then they were digested in a solution of HClO4-HNO3 (Armentia et al., 2008). Pb, Cd, and Mg concentration were measured by atomic absorption spectrophotometry in a PerkinElmer PinAAcle 900 F (detection limit 0.05 mg/L) following the method SW-846 7000 (EPA, 2003). Arsenic was measured by hydride generation in an external certified lab, with a detection limit of 0.005 mg/L. All the analyses were done in duplicates.

The presence of total coliforms and Salmonella spp was assessed, adapting locally developed protocols (Castañeda, 2004; PPTAR, 2014). Sieved soil and whole grains were suspended in sterilized casein peptone solution, and they were then mixed in a vortex. Tenfold dilutions were filtered and added to Petri dishes containing BBL (Salmonella) or Difco mFC (coliforms) agar, to be incubated for 24 h at 35 ± 2 °C (coliforms) and 48 h at 35 ± 2 °C (Salmonella). Deionized sterilized water and a solution containing both pathogens were used as negative and positive controls. After incubation emergence of colonies was assessed by direct inspection. All microbial tests were done in triplicates.

### Figure 1.
SEM images from struvite obtained in this research.

3. Results and discussion

#### 3.1. Characterization of struvite

A yield of 27.92 kg struvite (dry basis) for each cubic meter of leachate was achieved. It had a specific gravity of 0.54 t/m³ on dry basis. The 86% of ammonia nitrogen and 89% of phosphorous was removed from leachate, achieving a final suspended solids concentration of 20.5 mg/L. The struvite obtained by precipitation of the leachate showed two crystalline forms: regular round shapes and long prisms, as shown in the SEM image (Figure 1). The observed shapes are similar to those obtained by treating the leachate of the Hong Kong landfill (Li and Zhao, 2003).

The identity of the struvite was confirmed by energy-dispersive X-ray spectroscopy analysis (Figure 2), which showed a typical profile of the mineral. This test also allowed us to identify the presence of potassium in regular crystals of struvite, which could improve the efficiency of struvite as fertilizer. In addition, the presence of other elements in the mineral can be related to the raw material used to prepare the mineral; previous research has shown that besides Mg, other nutrients such as Ca, K, Na, and Fe can be present in struvite (Uysal et al., 2014).

The concentration of heavy metals found in the obtained mineral is similar to the one reported, showing a higher level of potassium and calcium. The molar relation obtained was 2Mg:1NH₄:1.2PO₄, similar to the one reported previously (Di Iaconi et al., 2010). However, the molar ratio of the obtained mineral differs from the stoichiometry of struvite (Mg:NP = 1:1:1), possibly due to the coprecipitation of struvite along with other minerals such as magnesium phosphate, calcium phosphate, and magnesium hydroxide. The elemental composition and its comparison with reported values are presented in the Supplementary material.

The NH₄PO₄ ratio obtained in struvite in this research (0.34:1) is lower than previously reported values of 0.43:1 (Di Iaconi et al., 2010) and 0.59:1 (Szymanska et al., 2019), while similar to the obtained by others (Uysal et al., 2014; Wramadewanthi et al., 2021). This ratio is the result of factors such as the crystallization process, the proportion of Mg, the pH, the aeration rate, the retention time, and the temperature (Rahman et al., 2014). It is also related to the initial molar ratio Mg:NH₄:PO₄ which was 2:1:1.2, different from previously used values of 2:1:1 (Di Iaconi et al., 2010) and 1.5:1:1 (Uysal et al., 2014).

The concentration of metals was lower when compared to phosphate fertilizers, which can include 2−1,200 mg As/kg, 7−225 mg Pb/kg, 7−179 mg Cd/kg, 1−12 mg Co/kg and 0.1−0.12 mg Hg/kg (Kabata-Pendias, 2011). These low metal content has been reported previously for struvite produced from the effluent of the yeast industry (Uysal et al., 2014), and landfill leachate (Li and Zhao, 2003). The metal content also complies with the maximum levels for the national regulations for biosolids (SEMARNAT, 2002) and soils intended for agriculture (SEMARNAT, 2004).

However, it must be noticed that presence of ionic species may hinder the efficient of struvite recovery, product purity, morphology, and reaction speed. High concentrations of Ca in particular are known to precipitate simultaneously as phosphate salts along struvite (Krishnamoorthy et al., 2021; Pastor, 2008), mainly at pH > 10. Soluble ions could also precipitate due to chelation into struvite (Nandhri et al., 2021; Wramadewanthi et al., 2021). Coprecipitation of other minerals, such as hydroxyapatite, sulfohalite, and trisodium dipotassium tripolyphosphosilicate has been reported before (Warmadewanthi et al., 2021), affecting the yield of struvite formation. While no further analysis was performed to identify specific minerals, it can be assumed that not all the obtained mineral was struvite, due to the presence of calcium.

#### 3.2. Harvest of maize and assessment of yields

The maize was harvested in December, six months after sowing. The complete process, from land preparation to tillage of stubble, is shown in Figure 3.
The production of maize when struvite was added as fertilizer was higher in two of the three sites (Figure 4). At La Mesa, 4,165.76 kg/ha were obtained, showing a yield of 6.36% higher than the commercial fertilizer and 8.38% than the control. A similar result was observed in La Tarjea, where the production with struvite (3,368.5 kg/ha) was 2.16% and 33.8% higher than the yield observed for the commercial fertilizer and the control, respectively. On the other hand, in Hierbabuena, the higher production was achieved in the control (4,059.84 kg/ha), probably due to the poor drainage conditions that lead to flooding in the plots where struvite and the commercial fertilizer were used.

Figure 2. EDS spectrum from irregular crystals (left) and long prisms (right).

Figure 3. Sowing and harvesting of maize process.

Figure 4. Production of maize (left) and biomass (right) for different locations and treatments.
In general, struvite showed a very competitive performance (Table 2), considering that it was applied in a lower dosage than the DAP + urea mixture (73.3% less than required). This result coincides with previous research, where struvite was compared to ammonium phosphate, showing similar or higher yields (Szymanska et al., 2019). Furthermore, the addition of fertilizers also increased the size of the grains. For example, the mass of 50 grains showed that in La Tarjea and La Mesa, fertilized soils increase up to 8.7% in the mass of the grains.

Production of biomass in La Mesa and La Tarjea was higher for the DAP + urea mixture than for struvite (6.22% and 0.83% for each site). This increase can be explained by a higher mass of stubble when the commercial product was used. In Hierbabuena, as it happened for maize, the yield was higher for the non-fertilized plot. The results of La Mesa and La Tarjea showed that while struvite increases maize productivity, the DAP + urea mixture increases the yield of stubble. The positive effect due to the addition of struvite coincides with previous research where maize, tomato, and grass showed higher biomass yield when compared to commercial formulae (Liu et al., 2011; Szymanska et al., 2019; Uysal et al., 2014). Li and Zhao (2003) found similar results for four fast-growing (Brassica parachinensis, Brassica rapa var. chinensis, Ipomoea aquatica, and Ipomea aquatica, I. reptans). They found that the overdosage of struvite (2–8 times) did not harm the growth of the plants due to their low solubility. When struvite was compared to mineral phosphate fertilizers in the growth of Lactuca sativa L., the plants rendered higher fresh biomass and P absorption, explained by the authors by a synergistic effect produced by the presence of Mg (González et al., 2009).

3.3. Effects of struvite in nutrient assimilation

The characterization of grain is shown in Table 3. The use of struvite did not affect the organoleptic characteristics of the maize. The level of nitrogen (10.50 ± 0.42 to 13.25 ± 0.78%) is close to previously reported values, which go from 14% in unfertilized tests to 15.1% in Zn-enriched soils in the interval commonly reported for maize (Puga et al., 2013). The lower levels obtained can be related to the initial quality of the soil (Kabata-Pendias, 2011). There was no clear tendency in the assimilation of N derived from the use of the fertilizers, as struvite showed better performance than the commercial mixture in one site and lower values in the others. This finding, different from previously reported results (Puga et al., 2013), can be attributed to the lower dosage of struvite applied when compared to the DAP + urea fertilizer. Despite the lower mass added, struvite also increased nitrogen content in the soil above the commercial mixture.

Content of phosphorous in maize has been reported in the intervals of 3.0 to 3.1 g/kg in Zn-enriched soils (Puga et al., 2013), and 2.9 g/kg in dry grain (Uhart and Echeverría, 1998). In this study, the grains obtained in plots where fertilizer was used showed a phosphorous concentration of 2.1 ± 0.05 g/kg, coincident with previously reported results. The maize without fertilizers (CTRL) assimilated less phosphorous; however, it is within limits described as expected (2.1 mg/kg) (TA, 2019). Other authors have found that struvite increases the assimilation of nitrogen and phosphorous when compared against commercial fertilizers (González et al., 2009; Puga et al., 2013; Szymanska et al., 2019; Uysal et al., 2014), the variable results obtained in this research can be attributed to the lower dosage of struvite applied when compared to the DAP + urea.
Salmonella spp. and Fecal coliforms were detected in the soils and grains of all experimental sites. The detection of these pathogens is a concern as they are considered indicators of fecal pollution, which can originate from various sources such as animal waste, human sewage, and agricultural activities. The presence of these pathogens can indicate a potential risk to human and animal health, as well as to the environment.

The study also highlights the importance of monitoring heavy metals such as Pb, As, and Cd in the soil and grains. The detection of Pb in the soil and grains of La Mesa and Hierbabuena, as well as As in La Tarjea, indicates that these metals may be sources of contamination. The detection of Cd in La Mesa and Hierbabuena suggests that this metal may also be a concern in these areas.

The study also provides valuable information on the nutrient content of the grain. The concentration of magnesium in maize grains ranged from 12.5 to 24.54 mg/kg in different experimental sites. This increase in the content of this nutrient in soil when using struvite can be attributed to the baseline concentration in the soil, which ranged from 12.5 to 24.54 mg/kg. In general, the nutrient content of the grain was higher when using struvite as a fertilizer, which suggests that the use of struvite can be an effective way to increase the nutrient content of crops.

The study also highlights the importance of considering the environmental impacts of using struvite as a fertilizer. The presence of Pb in the soil only was detected in the traditional fertilized plot in La Mesa, the control, and the struvite plots in La Tarjea. In all cases, this can be attributed to the baseline concentration in soil, which ranged from 12.5 to 24.54 mg/kg. The neglectable contribution of Pb from struvite has been reported before (Uysal et al., 2014). The regulation mentioned above sets maximum levels of 22 mg/kg for As and 37 mg/kg for Cd. These metals were not found in the grains, and their level in soils, in all cases below the limits, can be considered as typical baseline concentrations (Kabata-Pendias, 2011).

Pathogens were found only in grains and soils of La Mesa. This finding is highly likely a consequence of the soil contamination, which could be caused by nearby cowsheds and the use of water from the river Santiago for irrigation. This river receives untreated wastewater discharges, as shown in the supplementary material, and has been assessed as highly polluted (IACHR, 2020).

3.4. Safety assessment of the use of struvite

One of the main concerns related to the use of landfill leachate to produce struvite is the possible transfer of pollutants, mainly heavy metals and pathogens, to the soil and plants. In Mexico, the legal regulation that fixes the sanitary limits for the production of grains, NOM-247-SSA1-2008 (SS, 2008), sets a maximum Pb content of 0.5 mg/kg. In this research, none of the experimental conditions exceeded that limit (Table 4). The presence of Pb in the soil only was detected in the traditionally fertilized plot in La Mesa, the control, and the struvite plots in La Tarjea. In all cases, this can be attributed to the baseline concentration in soil, which ranged from 12.5 to 24.54 mg/kg, into the allowed range established in the Mexican norm for soil intended for agriculture (400 mg/kg) (SEMARNArt, 2004). The obtained struvite has less than 0.500 mg/kg of Pb, and no correlation was observed related to its use.

The production cost for struvite in this research was 629.8 USD/ton. This cost is high when compared to reported prices for struvite (Table 5). Compared with current alternatives in Mexico also shows a higher cost when using struvite; while struvite had a total cost, considering the applied dosage, of 1370 USD/ha, the cost of DAP + urea was 271 USD/ha. However, the additional benefits of using struvite must be taken into account: first, it allows recovering nutrients from solid and liquid wastes. Second, it is a more complex slow-release fertilizer, contributing to primary and secondary nutrients, such as N, P, K, Mg y Ca, while DAP is applied as a commercial mixture DAP + urea.
mainly a nitrogen source. As struvite needs only one application, its use can lead to lower farming costs.

The potential production of struvite from landfill leachate in the state of Jalisco, based on a daily generation of 5000 t/day with 30% moisture, is 41.8 kg/day. However, yield and selectivity could be improved by the optimization of the operation parameters. Other alternatives of this source consist of leachate wastes from nearby farms (Darwish et al., 2017) and residual phosphoric acid. On the other hand, the use of cheaper Mg sources, such as MgO (Huang et al., 2014; Stolzenburg et al., 2015), MgSO₄, MgCl₂ (Di Iaconi et al., 2010) or even seawater could be assessed (Shin and Lee, 2010). Additional options to decrease the cost of the process include pretreatment to eliminate ions such as Ca and increasing the yield to struvite (Warmadewanthi et al., 2021), recycling of struvite in more than one cycle (Huang et al., 2014), or its application as the first stage on a treatment train. It should be noticed that the production of struvite would substitute current technologies used for leachate treatment, such as reverse osmosis, whose cost has been estimated as US$ 8.58/m³ (Almeida et al., 2020). In the region, the cost of leachate treatment ranges from US$ 15/m³ to US$ 30/m³; the production of 1 t of struvite would eliminate the cost of treatment from approximately 35 m³ of leachate, which would cost US$525– US$1059. This avoided treatment cost improves the economic feasibility of the process.

4. Conclusions

This research shows the feasibility of the recovery of nutrients from landfill leachate for their use as fertilizer in the production of maize. Struvite, the obtained fertilizer, showed a similar performance to the commercial control, even if it was applied in lower soil:fertilizer proportions. The results are promising, as the process would contribute to the recycling and conservation of natural resources.

Leachate can include different pollutants coming from waste that could transfer to soil and maize through the use of struvite. Nevertheless, no adverse effects were observed regarding pollutants transfer in this field test. However, it must be considered that the composition of leachate is inherently variable, so continuous monitoring would be required to guarantee the safety of struvite in terms of possible migration of metals, pathogens, and other contaminants. Another issue to be solved is the reduction in the production cost of struvite, which currently could be seen as a limitation if the product is commercialized and compete with fertilizers currently in the market. However, the equilibrium point could be reached if those costs and the externalities related to the current leachate management are considered.

On the other hand, the process could render different environmental benefits, such as diminishing the need to extract nutrients to produce fertilizers, the decrease of the nutrients load in the leachate, and their reincorporation into the production cycles. This alternative would be especially attractive to countries like Mexico, where the burial of organic waste in landfills is still a common practice and where farming is also practiced in a non-industrialized way.

Declarations

Author contribution statement

Deborah Lucero-Sorbazo: Conceived and designed experiments; Performed the experiments; Contributed reagents, materials, analysis tools or data.
Margarita Beltrán-Villavicencio: Performed the experiments; Contributed reagents, materials, analysis tools or data; Analyzed and interpreted data; Wrote the paper.
Abealdo González-Aragón: Analyzed and interpreted data.

Alethia Vázquez-Morillas: Analyzed and interpreted data; Wrote the paper.

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Data included in article/supp. material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

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