

BULETINUL INSTITUTULUI POLITEHNIC DIN IAȘI

Publicat de

Universitatea Tehnică „Gheorghe Asachi” din Iași

Volumul 70 (74), Numărul 4, 2024

Secția

CHIMIE și INGINERIE CHIMICĂ

DOI: 10.5281/zenodo.14582031

EFFECT OF INORGANIC SALTS ON THE SOLUBILIZATION OF PHOSPHORUS FROM CHEMICAL FERTILIZERS

BY

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Received: September 10, 2024

Accepted for publication: November 26, 2024

Abstract. In this study, the effect that inorganic salts have on the solubilization of phosphorus from chemical fertilizers was analyzed. A commercial fertilizer (NKP = 15% - 15% - 15%) and the inorganic salts CuSO₄, CaCO₃ and CuCl₂ were used for the experimental studies. All analyzed samples were obtained by mechanical mixing a constant amount of solid fertilizer with 1 – 2% inorganic salts, followed by their dissolution in distilled water. The obtained results showed that the presence of inorganic salts significantly affects the solubilization of phosphorus, the decrease in the concentration of soluble phosphorus in the obtained solutions being 14.5 – 29.6% lower than in the control sample. An important decrease in the concentration of easily soluble phosphorus can be also observed when fertilizer samples were placed in the soil. The presence of inorganic salts leads to a decrease in the concentration of soluble phosphorus in the soil samples by up to 54% compared to the control sample. These results highlight the effect that inorganic salts have on the efficiency of the use of phosphorus chemical fertilizers to increase soil quality.

Keywords: chemical fertilizer, phosphorus solubilization, inorganic salts, soil quality.

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1. Introduction

Phosphorus is an indispensable macro-element for the growth and development of cultivated plants, which intervenes in numerous metabolic processes (ex. the metabolism of carbon, phospholipids, nucleic acids, etc.) (Dawson and Hilton, 2011; Lambers, 2022), thus facilitating the regulation of enzymatic activity at the level of plant cells (Li *et al.*, 2015; Langhans *et al.*, 2022). Therefore, ensuring the optimal phosphorus content in agricultural soils is particularly important, and where this is not achieved naturally, fertilizers are used. This is why the production of phosphorus-based fertilizers is continuously increasing, reaching over 47 million metric tonnes globally in 2023-2024 (Ahash *et al.*, 2024).

To determine whether or not fertilizers use is necessary, as well as the amount of fertilizer needed, continuous monitoring of the phosphorus content is particularly important from an agrochemical point of view. Unfortunately, the determination of the total phosphorus content is not always sufficient to assess the quality of soils used for growing plants.

Due to its chemical properties (Jones *et al.*, 2019; Hou *et al.*, 2020), phosphorus can be involved in numerous chemical equilibria that easily occur under soil conditions, forming chemical compounds with low solubility (insoluble compounds). Such undesirable chemical equilibria are favoured by the presence of inorganic or organic ions/molecules, which are abundant in the soil. Due to their presence in the soil, phosphorus mobility decreases significantly and may become unavailable to plants due to some secondary adsorption and/or precipitation processes, which leads to a decrease in fertilizer efficiency. Studies in the literature (Tilman *et al.*, 2011; Yu *et al.*, 2021) have shown that of the total amount of phosphorus added to the soil using fertilizers, less than 25% is accessible to plants during a growing cycle. All these aspects must be taken into account when calculating the amount of fertilizer required for fertilization, and to achieve this it is necessary to analyze the amount of phosphorus that dissolves from the fertilizer and becomes available to plants.

As expected, the concentration of soluble phosphorus will depend on the type of soil and the type of fertilizer (Heuer *et al.*, 2017; Oliveira *et al.*, 2019). Any change in the fertilizer production process (use of a different raw material, change in operating conditions, addition of new additives, etc.), can cause a change in the amount of soluble phosphorus, and thus significantly influence the efficiency of the fertilizer. Therefore, it is necessary to periodically analyze the concentration of soluble phosphorus in the fertilizers, but also in the soil.

Some of the most common fertilizers used to correct the phosphorus content of the soil are NPK fertilizers (nitrogen-phosphorus-potassium fertilizers) (Eddarai *et al.*, 2022; Phares and Akaba, 2022). The widespread use of these types of fertilizers is due to: (i) their easy manufacture, which involves simply mixing the three components (potassium chloride, ammonium nitrate and ammonium

phosphate) in liquid or solid phase (Van Vuuren *et al.*, 2010), (ii) the wide availability of raw materials (phosphate and nitrate rocks are easy to extract and can be found in many region of the world, or can be replace by industrial wastes) (El Bamiki *et al.*, 2021), and (iii) their high efficiency in increasing soil quality. Therefore, finding new types of raw materials and improving the quality of NPK fertilizers is a constant concern of industrial research in this field.

The addition of microelements essential for plant growth to the composition of NPK fertilizers is one of the research directions aimed at improving the efficiency of these fertilizers in agricultural uses (Wyszkowski and Brodowski, 2020; Yadav *et al.*, 2022). Unfortunately, increasing the concentration of inorganic ions (due to the addition of microelements) can lead to a significant variation in the concentration of soluble phosphorus in the NPK fertilizer, due to the adsorption/precipitation equilibriums. Therefore, the nature of the inorganic salt used as a microelement additive and its concentrations play an important role in assessing the concentration of soluble phosphorus that can reach the soil.

In this study, four types of mixtures were prepared, starting from NPK 15-15-15 fertilizer (NPK-0), and using as microelement additives the inorganic salts: 1% CuSO_4 (NPK-1), a mixture of 1% CuSO_4 and 1% CaCO_3 (NPK-2), and 2% CuCl_2 (NPK-3), respectively. For each case, the concentration of soluble phosphorus, both in distilled water and soil, was determined. The obtained results allow a much more precise assessment of the amount of fertilizer that must be used in agricultural soils to ensure the desired concentration of phosphorus.

2. Materials and methods

2.1. Materials

Commercial NPK 15-15-15 fertilizer (NPK-0) purchased from the local market was used for the experimental studies. Analytical grade inorganic salts (CuSO_4 , CuCl_2 and CaCO_3) (purchased from Chemical Company, Romania) were used without further purification. Three fertilizer mixtures, namely: NPK + 1% CuSO_4 (NPK-1), NPK + 1% CuSO_4 + 1% CaCO_3 (NPK-2) and NPK + 2% CuCl_2 respectively, were prepared and used in the laboratory studies. A solution of $13.58 \text{ mg P}\cdot\text{L}^{-1}$, prepared by dissolving an appropriate amount of K_2HPO_4 , was used to plot the calibration curves. The soil sample used in the experimental studies was taken from the vicinity of “Gheorghe Asachi” Technical University of Iași, and after drying (in air at room temperature) and homogenization, it was subjected to preliminary tests.

2.2. Experimental procedures

The experimental studies were carried out in two stages. In the first stage, 2 g of each fertilizer (NPK-0, NPK-1, NPK-2 and NPK-3) was dissolved in 50

mL of distilled water, until clear solutions were obtained. All solutions were left to stand for 24 hours (to reach the equilibrium), after which they were used to determine the phosphorus concentration, pH and electrical conductivity. In the second stage, 0.2 g of each fertilizer (NPK-0, NPK-1, NPK-2 and NPK-3) was placed in top of 5 g of soil, in plastic containers (5 x 5 x 5 cm), with drainage holes at the bottom. Each sample was watered daily with 1 mL of distilled water. A series of 5 samples was prepared for each type of fertilizer so that the soil could be watered for 5 days. After 24 hours of each watering, the fertilizer was removed, and soil samples were analyzed. The parameters measured for these samples were: moisture, soluble phosphorus concentration, pH and electrical conductivity.

2.3. Analytical methods

In the case of aqueous solutions: The soluble phosphorus concentration was analyzed spectrophotometrically (Digital Spectrophotometer S 104D), using ammonium molybdate as a colour reagent ($\lambda = 650$ nm/against blank solution, 1-cm glass cell). pH was determined with a Mettler Toledo pH/ion meter, equipped with a combined glass electrode. The electrical conductivity ($1/R$ [$\mu\text{S}\cdot\text{cm}^{-1}$]) was measured with a Jenwa-4590 conductometer, equipped with a conductometric cell (electrodes area = 1 cm^2). In the case of soil samples: Moisture was analyzed gravimetrically, according with the standard methodology (105°C , 6 hours). For the determination of the other parameters (soluble phosphorus concentration, pH and electric conductivity), 2 g of each soil sample was mixed with 10 mL of distilled water and stirred continuously for 1 hour. After filtration, the obtained solutions were used for the analytical measurements, using the methods described above.

3. Results and discussion

3.1. Determination of soluble phosphorus concentration in aqueous solution

All fertilizer samples (NPK-0, NPK-1, NPK2 and NPK-3) dissolved relatively easily in distilled water, with complete dissolution (clear solutions) within 30 min. However, after allowing the solutions to stand for 24 hours, white solid particles were observed to appear at the bottom of laboratory flasks. This indicates that secondary precipitation (even adsorption) equilibriums occur in aqueous fertilizers solutions, which can modify the concentration of soluble phosphorus. Under these conditions, it is necessary to accurately analyze the concentration of soluble phosphorus in each solution to evaluate the efficiency of the fertilizers.

In Fig. 1, the values of soluble phosphorus concentration, pH and electrical conductivity ($1/R$), obtained for each fertilizer (NPK-0, NPK-1, NPK-2 and NPK-3) are presented.

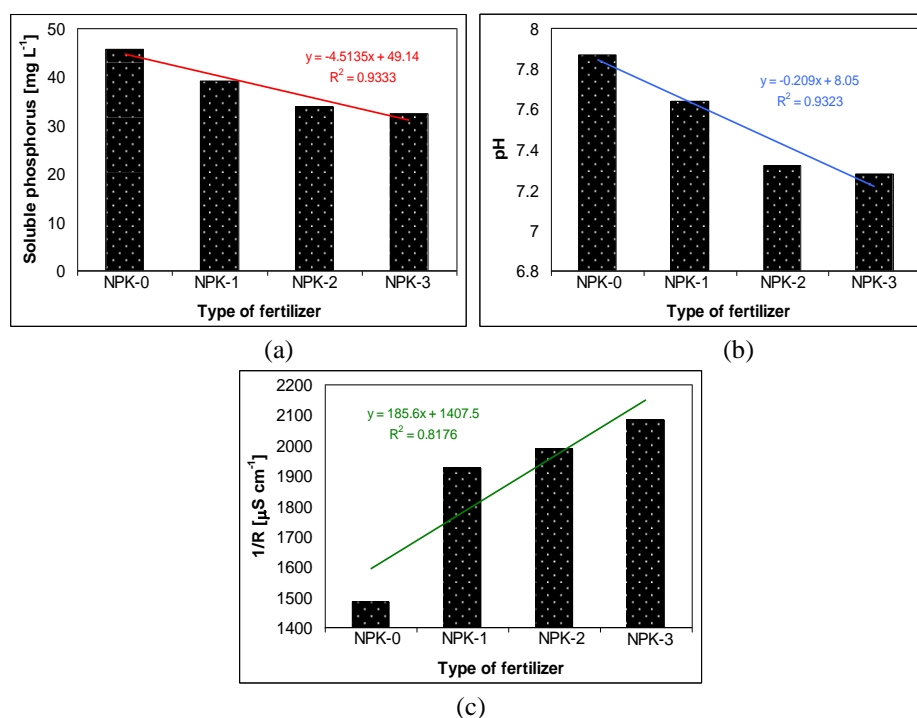


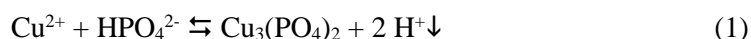
Fig. 1 – Variation in soluble phosphorus concentration (a), pH (b) and electrical conductivity (c) depending on the type of fertilizer.

As can be seen from Fig. 1a, the presence of salt additives leads to a decrease in the concentration of soluble phosphorus in the fertilizer. The smaller decrease is recorded in the case of NPK-1 (14.5% compared to NPK-0), while the largest decrease corresponds to NPK-3 (29% compared to NPK-0). A similar decreasing trend can be observed and in the case of pH (Fig. 1b), even though the decrease in the values of this parameter in the presence of salt additives is below 0.6 units compared to the initial fertilizer (NPK-0). Unlike the concentration of soluble phosphorus and pH, in the case of electrical conductivity, the presence of salt additives in the fertilizer composition leads to an increase in the values of this parameter, as can be seen in Fig. 1c. This increase is almost 30% in the case of NPK-1, but reaches over 40% in the case of NPK-3, compared with the fertilizer sample without salt additives (NPK-0).

All these experimental results show that adding inorganic salts to increase fertilizer efficiency can lead to a decrease in the concentration of soluble

phosphorus, which is important for plant growth. Taking into account the variation in pH (Fig. 1b) and electrical conductivity (Fig. 1c), this decrease in soluble phosphorus concentration is most likely due to secondary precipitation equilibriums, caused by the presence of salt additives.

Thus, the presence of Cu(II) ions in the composition of NPK-1, NPK-2 and NPK-3 fertilizers allows the formation of less soluble compounds $\text{Cu}_3(\text{PO}_4)_2$ ($K_w = 1.3 \cdot 10^{-37}$) (Dean, 1995), according with the equilibrium:



Achieving this precipitation equilibrium leads to the release of H^+ ions, which explains the experimentally observed decrease in pH (Fig. 1b). As a result, part of soluble phosphorus generated by the dissolution of the fertilizer (which exists in solution in the HPO_4^{2-} speciation form, at this pH value) is consumed, and its concentration decreases. The even more pronounced decrease in the concentration of soluble phosphorus in the case of NPK-2 and NPK-3 fertilizers compared with NPK-1 is determined by the simultaneous achievement of adsorption and precipitation equilibriums. Thus, the presence in NPK-2 of CO_3^{2-} ions (ions with high hydration energy ($\Delta G_{\text{hyd}} = -1315 \text{ kJ/mol}$) (Marcus, 1991)) causes the number of free water molecules to decrease significantly, which will favour the precipitation of $\text{Cu}_3(\text{PO}_4)_2$. In addition, the formation of CuCO_3 precipitate cannot be excluded, although its solubility is much higher ($K_w = 9.8 \cdot 10^{-10}$), compared with $\text{Cu}_3(\text{PO}_4)_2$ ($K_w = 1.3 \cdot 10^{-37}$) (Dean, 1995). Consequently, the concentration of soluble phosphorus decrease even more, compared to NPK-1 (by 13.5%). In the case of NPK-3, the use of CuCl_2 as an additive is responsible for the appearance of solid particles, on which $\text{Cu}_3(\text{PO}_4)_2$ can be easily adsorbed (or co-precipitated). This is because it is well known that CuCl_2 can hydrolyze easily, and upon hydrolysis a basic salt ($\text{Cu}(\text{OH})\text{Cl}$) is formed, which is poor soluble. The solid particles formed upon hydrolysis of CuCl_2 behave as an adsorbent material, on the surface of which ions or molecules from aqueous solution can be retained. These secondary equilibriums make the concentration of soluble phosphorus in NPK-3 fertilizer much lower (over 28%) compared with NPK-0 fertilizer.

3.2. Determination of soluble phosphorus concentration in soil samples

The results obtained when determining the moisture content of the soil samples are shown in Fig. 2. Although the moisture values increase with the increase in the number of watering days, there are no major differences between the 4 soil samples ($\pm 10\%$). Therefore, the measured values for the other experimental parameters (soluble phosphorus concentration, pH and electrical conductivity) are practically not influenced by the total amount of distilled water

used for to water the soil samples, but only by the elementary equilibriums (dissolution, precipitation, adsorption) that take place in the soil mass.

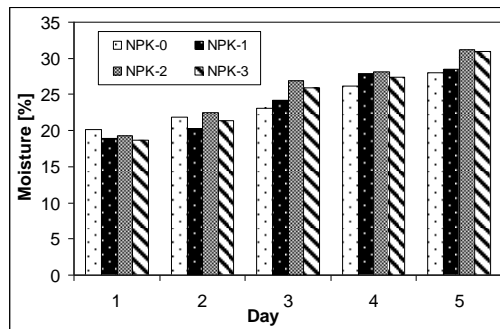


Fig. 2 – Variation in soil sample moisture during of watering.

Figure 3 illustrated the variation in soluble phosphorus concentration for each of the 4 soil samples, during the 5 days of watering. Analyzing the experimental data presented in Fig. 3, two observations can be made: (i) for NPK-0 and NPK-1 fertilizers, the soluble phosphorus concentration in soils sample increases continuously with the number of watering days, throughout the experimental period, and (ii) for NPK-2 and NPK-3 fertilizers, the soluble phosphorus concentration decreases significantly after first day of watering, although a slow increase in this parameter can be observed on days 2-5.

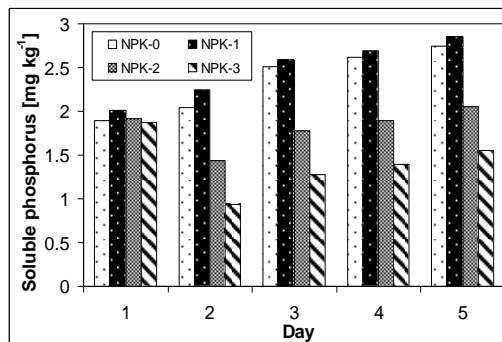


Fig. 3 – Soluble phosphorus concentration in soil samples for each of the 5 days of watering.

The continuous increase in soluble phosphorus concentration observed for soil samples containing NPK-0 and NPK-1 fertilizers suggest that as the number of watering operations increases, these fertilizers dissolved more and more, releasing phosphorus that reaches the soil and can contribute to plant growth. In the case of the studied soil, it is likely that Cu(II) ions resulted from

the dissolution of the additive (CuSO_4 , for NPK-1) easily bind to the organic matter in the soil and have a minimal effect of phosphorus solubility. Consequently, the soluble phosphorus concentration in soil sample containing NPK-1 fertilizer increase after each watering, and the differences compared to the soil sample containing NPK-0 fertilizer are quite small (3 – 10%) (Fig. 3). In contrast, in the case of soils samples containing NPK-2 and NPK-3 fertilizers, the concentration of soluble phosphorus decrease significantly after first day of watering, with differences ranging between 25 – 29% for NPK-2 and 43 – 54% for NPK-3 respectively, compared to NPK-0 (Fig. 3).

This significant decrease shows that in the case of soil samples containing NPK-2 and NPK-3 fertilizers, phosphorus is involved in secondary processed, most likely precipitation, generated by the presence of salt additives. Thus, in the case of NPK-2 fertilizer, the presence of Ca(II) ions reduces the possibility of binding Cu(II) ions to the organic matter of the soil. This makes the Cu(II) ions resulting from the dissolution of the additives remain available and can interact with the dissolved phosphorus (HPO_4^{2-} ions) forming a precipitate. Therefore, as the number of watering operations increase, the amount of dissolved Ca(II) and Cu(II) ions increases, and the concentration of soluble phosphorus decreases. In the case of NPK-3 fertilizer, the decrease in the soluble phosphorus concentration is most likely a consequence of the hydrolysis of CuCl_2 used as an additive. Adding the water leads to the formation of hydrolysed salt (Cu(OH)Cl), and creates the necessary conditions for the phosphorus dissolution equilibrium to be retrograded.

These assumptions are supported by the pH and electrical conductivity values measured during the 5 days of watering, for each soil sample (Fig. 4).

Although pH does not vary significantly, all values being between 6.8 and 7.5, an increase in the values of this parameter can be observed with the increase in the number of watering days (Fig. 4a), for all analyzed soil samples. This supports the hypothesis of the existence of secondary equilibriums accompanying the dissolution of fertilizer (NPK-0) or the mixture of fertilizer and salt additives (NPK-1, NPK-2 and NPK-3).

On the other hand, the much clearer variation in the electrical conductivity (Fig. 4b) shows that these secondary equilibriums are most likely precipitation processes. Thus, if in the case of soil samples containing NPK-0 and NPK-1 the occurrence of precipitation equilibriums is limited (electrical conductivity increase with increasing number of watering days, which suggest that more and more ions are passed into the soil solution), in the case of soil samples containing NPK-2 and NPK-3, precipitation equilibriums have a significant share.

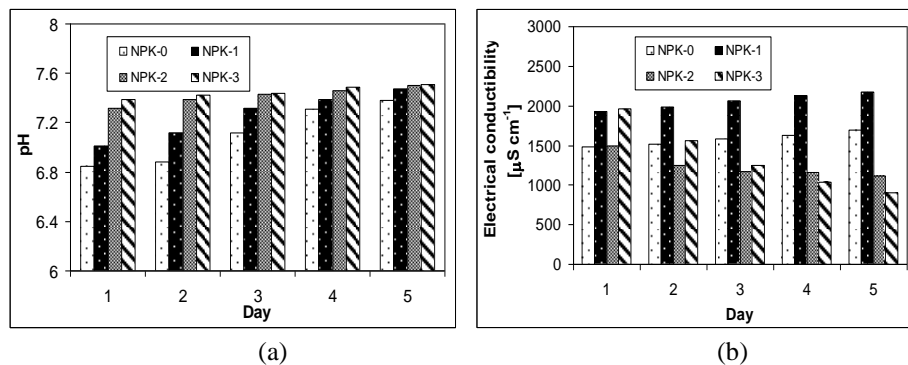


Fig. 4 – Variation of pH (a), and electrical conductivity (b) for soil samples during of watering.

Due to these, the electrical conductivity of these soil samples decreases with increasing number of watering days (Fig. 4b), which shows that once dissolved, the ions pass into an un-dissociated (precipitate) form. The possibility of secondary equilibriums occurring when using mixture of fertilizers and additives must be detailed analyzed and taken into account when calculating the amount of fertilizer that must be administrated to an agricultural crop to obtain the desired yield.

3. Conclusions

In this study, the effect that inorganic salts, have on the solubilization of phosphate from NKP = 15% - 15% - 15% fertilizer, both in aqueous solution and in soil, was examined. Three fertilizer mixtures, namely: NPK + 1% CuSO_4 (NPK-1), NPK + 1% CuSO_4 + 1% CaCO_3 (NPK-2) and NPK + 2% CuCl_2 respectively, were prepared and used in the experimental studies. In aqueous solution the presence of salt additives leads to a decrease in the concentration of soluble phosphorus by 14.5% for NPK-1, by 26.2% for NPK-2 and by 29.6% for NPK-3 compared to NPK-0. Also, the measured values of pH and electrical conductivity suggest the occurrence of secondary precipitation equilibriums. In the case of soil samples, the soluble phosphorus concentration increases after each watering in the case of the sample containing NPK-1, while in the case of samples containing NPK-2 and NPK-3, a significant decrease in the soluble phosphorus concentration after the first day of watering, can be observed. And in the case of soil samples, the occurrence of secondary processes was highlighted using measured values for pH and electrical conductivity. All of these aspects must be taken into account when calculating the amount of fertilizer that must be administered to an agricultural crop to obtain the desired yield.

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EFFECTUL SĂRURILOR ANORGANICE ASUPRA SOLUBILIZĂRII FOSFORULUI DIN ÎNGRĂȘĂMINTELE CHIMICE

(Rezumat)

În acest studiu a fost analizat efectul sărurilor anorganice asupra solubilizării fosforului din îngrășămintele chimice. Pentru studiile experimentale s-a folosit un îngrășământ comercial (NKP = 15% - 15% - 15%), și sărurile anorganice CuSO_4 , CaCO_3 și CuCl_2 . Toate probele analizate au fost obținute prin amestecarea mecanică a unei cantități constante de îngrășământ solid cu 1 – 2% săruri anorganice, urmată de dizolvarea acestora în apă distilată. Rezultatele obținute au arătat că prezența sărurilor anorganice afectează semnificativ solubilizarea fosforului, scăderea concentrației de fosfor solubil în soluțiile obținute fiind de 14,5 – 29,6 % comparativ cu proba martor. O scădere semnificativă a concentrației de fosfor solubil poate fi observată și atunci când probele de fertilizator au fost adăugate în sol. Prezența sărurilor anorganice duce la o scădere a concentrației de fosfat solubil în probele de sol cu până la 54% față de proba martor. Aceste rezultate evidențiază efectul pe care sărurile anorganice îl au asupra eficienței utilizării îngrășămintelor chimice cu fosfat pentru îmbunătățirea calității solului.