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Article

# Potassium Citrate Nanoparticles in Zeolite Particles Improve the Properties of Acidic Soils and Increase Corn (*Zea mays* L.) Yield

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## Abstract

Potassium nanofertilizers on zeolite particles improve acidic soil conditions and increase maize yields. In this study, potassium citrate nanoparticles were synthesized and impregnated onto zeolite particles. The nanofertilizers and zeolite were analyzed using X-ray diffraction spectrometry, scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR). The efficacy of the materials was evaluated in acidic soils cultivated with maize. Two factors were considered for the evaluation—nanofertilizers (Nf1, Nf2, Nf3, and Nf4) and application time (T1 and T2)—resulting in eight treatments and one control, three replicates each (27 experimental units). The experimental plot contained two soil types; therefore, a randomized block design was used. The results confirmed differences between materials and increases in the potassium content of the impregnated zeolite. Treatment Nf3, applied after 40 days, increased pH and exchangeable potassium, resulting in increased corn grain yield per plant (216.90 g). These results suggest that potassium citrate nanoparticles impregnated with clinoptilolite zeolite act as a slow-release potassium nanofertilizer, representing a complementary alternative for fertilizing crops in acidic soils.

**Keywords:** *Zea mays* L.; nanomaterials; fertilizer; vertisols; regosols

## 1. Introduction

Nanofertilizers (Nfs) are composed of particles smaller than 100 nm in diameter. They are an alternative treatment for adding essential nutrients to the soil, so that plants can absorb them efficiently [1,2]. Likewise, the application of Nfs has been linked to an increase in plant growth-promoting substances in the soil [3] and higher crop yields [4].

Nfs can be produced through chemical (bottom-up) or physical processes (grinding) [5], ensuring the nanometric size of the particle [6], and can be of organic origin, such as chitosan [7], or mineral origin, such as silicate clays from the zeolite group, among others [8]. Currently, Nfs have been synthesized from a combination of organic substances and chemicals using the sol-gel process [9], such as in the case of iron citrate [6], where iron nitrate is combined with citrate to obtain iron nanoparticles [10].

Considering the above and the importance of potassium as an essential element for plants [11], which is a monovalent cation, the synthesis of potassium-based nanoparticles is plausible. In other words, potassium citrate nanofertilizer could be obtained. However, this type of compound is highly

soluble in water [12], which means that upon contact with soil moisture (soil solution), potassium could be rapidly lost through percolation processes [13]. This is why potassium citrate is applied as a foliar fertilizer to plants [14]. Therefore, it is important to prevent the rapid decomposition of potassium citrate and, in turn, the movement of potassium into the soil system.

Moreover, mineral-based nanoparticles are made with clinoptilolite zeolites (potassium zeolites), which are the most widely used nanoparticles in agriculture, due to their lower sodium content and high potassium concentrations. Therefore, they can improve the chemical conditions of the soil where they are added; for example, the pH of the soils [15]. Clinoptilolite can be reduced to nanometric sizes through milling or sonication processes. The main characteristic of zeolites is their porosity, which does not change with size changes; rather, their specific surface area increases [16]. These particles also serve as natural carriers of compounds or chemical substances, and even microorganisms in the soil [17,18]. Furthermore, research has indicated that crystals of chemical compounds can develop within the voids or pores of the zeolite after the zeolite particles are impregnated with solutions of these compounds and then dried [19].

Based on the above, we hypothesize that if a solution with a potassium citrate nanofertilizer were used to impregnate clinoptilolite particles and subsequently dry them, nanometric crystals of potassium citrate would be found on the surface of the zeolites in such a way that the zeolites would carry potassium citrate nanoparticles; then, when added to the soil, they would decrease the loss of  $K^+$  via percolation and increase the pH in acidic soils. In addition, potassium would be available at the crop's stage of greatest demand, which would be reflected in its yield.

If our hypotheses are proven correct, zeolite particles impregnated with potassium citrate nanofertilizers would represent an alternative treatment for the recovery or management of acid soils for corn (*Zea mays* L.) cultivation. Therefore, this work aimed to determine the physical and chemical characteristics of particles impregnated with potassium citrate nanofertilizer and evaluate the effect of applying these particles to soils with acidity problems, as well as their influence on the yield of corn during two crop cycles.

## 2. Materials and Methods

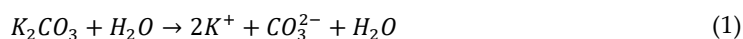
This study was conducted in three stages. The first stage was performed in the laboratory, where the nanofertilizer (Nf) and zeolite particles were obtained, and the materials obtained were characterized in the second stage. Finally, the third stage consisted of evaluating the materials in the field with corn crops to establish their effect.

### 2.1. Nanofertilizer Synthesis

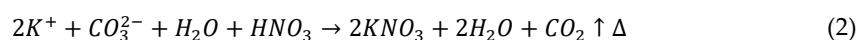
#### 2.1.1. Synthesis of Potassium Citrate Nanofertilizer

The Nf was synthesized using the low-temperature sol-gel (nitrate-citrate) process proposed by Polla [10] and Ghiasi [20] with some modifications. In this case, the reaction was performed in an aqueous medium (double-distilled water), with the variation being the addition of potassium carbonate (99% purity  $K_2CO_3$ , JALMEK) instead of calcium carbonate, followed by nitric acid (69% purity  $HNO_3$ , JALMEK) as an oxidant. Subsequently, citric acid (99.5% purity  $C_6H_8O_7$ , Merck) was added as a reducing and complexing agent to finally obtain potassium citrate ( $K_3C_6H_5O_7$ ), as shown in the following sequence of reactions:

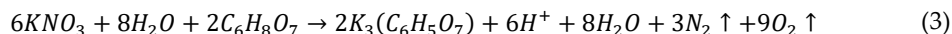
- Potassium carbonate undergoes dissolution or hydrolysis (Reaction 1).



- Potassium nitrate forms. When nitric acid ( $HNO_3$ ) is added to the system, an acid-base reaction occurs, where the nitric acid neutralizes the potassium carbonate, generating carbon dioxide ( $CO_2$ ) as a gas, water ( $H_2O$ ), and potassium nitrate ( $KNO_3$ ) (Reaction 2).



- The complex with citric acid forms. Citric acid ( $C_6H_8O_7$ ) combines with potassium ions ( $K^+$ ) present in the potassium nitrate ( $KNO_3$ ) solution, forming a potassium citrate complex [ $K_3(C_6H_5O_7)$ ] in an aqueous medium (Reaction 3).



- Citric acid has three carboxyl groups ( $-COOH$ ) that can react with  $K^+$  ions to form potassium carboxylates, keeping the base in the center of the citrate and thus making it a potassium citrate. This results in a solution with potassium citrate nanoparticles (Nf1) and potassium oxide, which was later used in the impregnation process and field tests.

### 2.1.2. Natural Zeolite

Zeolite particles were obtained from the San Rafael company, a mine located in San Luis Potosí, Mexico. Table lists the mineralogical and chemical characteristics of this material.

**Table 1.** Mineralogical and chemical characteristics of the clinoptilolite zeolite from this study.

Compound	Quantity
Clinoptilolite zeolite (Purity %)	85
Dolomite, feldspars, silicate clay and crystalline silica (%)	< 2
pH	8.1
CEC ( $cmol(+) kg^{-1}$ )	15
SiO <sub>2</sub> (%)	33.2
Al <sub>2</sub> O <sub>3</sub> (7%)	7.17
CaO (%)	1.91
K <sub>2</sub> O (%)	3.05
MgO (%)	0.56
Fe <sub>2</sub> O <sub>3</sub> (%)	1.4
Na <sub>2</sub> O (%)	0.70

Preparation. The commercial zeolite particles were of varying sizes, so they were homogenized using a set of sieves, obtaining two particle sizes. The first size had a diameter ranging from 2.0 to 2.5 mm, which was selected because it retained a greater amount of compounds [21] and is therefore recommended for zeolite impregnation. It can also be used as a carrier for smaller particles or nanoparticles [22]. Additionally, particles with a diameter smaller than 0.5 mm were also obtained. This particle size was selected because it can be applied directly to the soil, as it has a larger active surface area [21]. For the purposes of our study, this size represented the natural zeolite (Nf2) to be used in field tests.

### 2.1.3. Granulated and Impregnated Zeolite

Zeolite with a diameter of 2.0-2.5 mm (granulated) was activated with 0.1 M hydrochloric acid [23] according to the method proposed by Ola et al. [24]. This activation consisted of removing impurities, leaving exchange sites free, and lowering the pH of the material in solution. At the end of the process, the particles were washed with distilled water and dried in an oven at 105 °C.

- Impregnation: The activated zeolite was impregnated with the potassium citrate nanoparticle solution as follows: activated zeolite and the solution containing the potassium citrate nanofertilizer were placed in a 500 ml Erlenmeyer flask in a 1:3 ratio (w/w).
- Sonication: The mixture of zeolite particles activated with potassium citrate nanofertilizer was subjected to an ultrasonic bath for 30 minutes in a GRANBO 1120 W equipment, at 40 KHz, at room temperature. Subsequently, the flasks containing the solution were left to rest for nine days

in an incubator (Novatech) at a constant temperature (35 °C). At the end of this period, the supernatant was separated from the zeolite particles via decantation, as explained in the following section. The particles were washed with distilled water and dried in an oven at 105 °C for three days until a constant weight was achieved. Zeolite particles with potassium citrate were then obtained, representing the zeolites impregnated with potassium citrate nanoparticles (Nf3) to be applied in the field.

#### 2.1.4. Filtering of a Mixture of Granulated Zeolite and Potassium Citrate

The excess solution from the zeolite impregnation process with potassium citrate was filtered using a Buchner funnel with Wanthman No. 4 filter paper and a vacuum pump, and then stored in a refrigerator. This filtered remnant solution (Nf4) was used for field testing.

#### 2.2. Physical and chemical characterization of materials

The physical and chemical characteristics determined for Nf1, Nf2, Nf3, and Nf4 included their shape, size, and chemical composition, which were obtained using scanning electron microscopy (SEM) and elemental analysis via X-ray dispersive spectrometry (EDS), using a JEOL JSM-6610V scanning electron microscope. Fourier transform infrared spectroscopy (FTIR) analysis was performed using PERKIN-ELMER FTIR-FRONTIER equipment to obtain a graphical spectrum and identify functional groups and the molecular composition of the nanofertilizers.

#### 2.3. Field Evaluation of Nanofertilizer Treatments in the Field

##### 2.3.1. Study Area

This study was conducted on a plot of land located in San Martín Hidalgo, Jalisco, Mexico, located between 20° 24' 44" and 20° 24' 48" N and 103° 51' 50" and 103° 51' 55" W at an average altitude of 1350 meters above sea level. The climate of the region is BS1hw(x), which corresponds to semi-warm dry with rainfall in summer [25]. The average annual temperature is 21.0 °C, with a minimum and maximum of 7.9 °C and 33.1 °C, respectively, while the average total annual precipitation is 911 mm [26]. Its geology consists of alluvial sediments, although there are acidic extrusive rocks in the higher parts of the municipality, as well as basalts and tuffs. The predominant soils are Vertisol and Phaeozem, as well as Regosol, Luvisol, Cambisol, and Leptosol [27]. Land use is primarily agricultural (71% of the area).

The plot where the study was conducted had two types of soils arranged in strips from east to west. The soils were identified as Vertisol and Regosol, according to the World Reference Soil Database [28] (Table 2).

**Table 2.** Physical and chemical characteristics of the soils of the study plot.

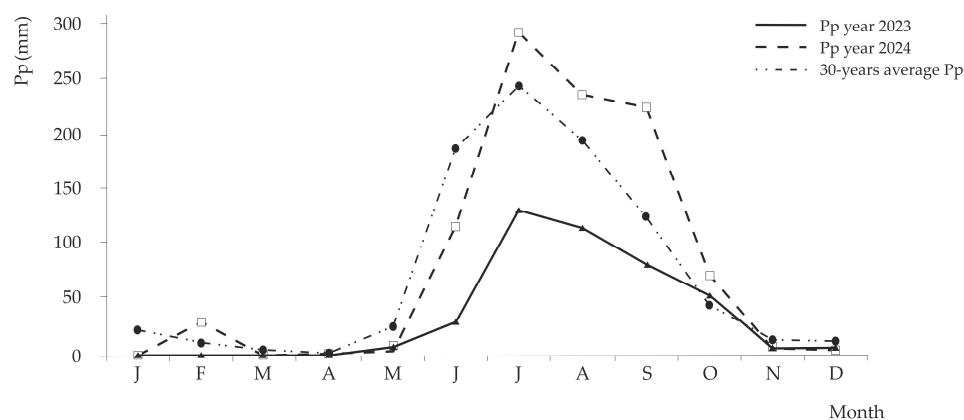
Parameters	Regosol	Vertisol	Reference
pH <sub>H2O</sub>	5.00 ± 0.40	4.58 ± 0.40	5.50 – 6.50
EC <sup>+</sup> (dsm <sup>-1</sup> )	1.46 ± 0.03	1.29 ± 0.01	1.00 – 2.00
OM (%)	3.40 ± 0.10	2.25 ± 0.10	2.50 – 3.50
Clay (%)	22.00 ± 1.50	38.00 ± 1.90	-
Lime (%)	14.00 ± 1.50	48.00 ± 2.50	-
Sand (%)	64.00 ± 2.90	14.00 ± 1.30	-
P (mg kg <sup>-1</sup> ) Bray	4.80 ± 0.40	11.00 ± 0.50	15.00 – 30.00

K(cmol(+) kg <sup>-1</sup> )	0.35 ± 0.01	0.54 ± 0.01	0.51 – 1.02
Ca (cmol(+) kg <sup>-1</sup> )	7.95 ± 0.05	13.60 ± 0.06	7.50 – 15.00
Mg (cmol(+) kg <sup>-1</sup> )	2.50 ± 0.02	3.60 ± 0.02	1.66 – 3.33
Na (cmol(+) kg <sup>-1</sup> )	0.61 ± 0.01	0.70 ± 0.01	0.21
CEC (cmol(+) kg <sup>-1</sup> )	13.70 ± 0.80	21.40 ± 0.90	12.00 – 25.00

<sup>†</sup>EC: electric conductivity; OM: organic matter; P: phosphorous; K: potassium; Ca: calcium; Mg: magnesium; Na: sodium; CEC: cation exchange capacity.

The tests were conducted over two years (summer, year 1 [year1]; summer, year 2 [year2]). The experiment in year1 was established on June 18, 2023, and on June 16, 2024, in year2. The corn hybrid used was Asgrow® Berrendo, a hybrid commonly planted in the region. The distance between rows was 70 cm, and 15 cm between plants. Note that the rows were oriented south–north, which did not change during the two years of the study. Cultural practices and crop fertilization were performed in accordance with the technology package for corn proposed for the region [29].

Weather conditions varied over the experimental period, especially precipitation (Figure 1). In 2023, total annual precipitation was 430.20 mm, resulting in a drought during the growing season (412.00 mm from June to October). In contrast, total rainfall in 2024 was 988.20 mm, with 941.00 mm during the crop cycle, which is more than double that of the previous year. The precipitation in 2024 falls within the range of 544.90 to 1092.10 mm obtained from a 30-year historical record [30]; this is considered normal, whereas the precipitation in 2023 was atypical.



**Figure 1.** Monthly precipitation (2023, 2024, and 30-year average) from San Martín Hidalgo, Jal. .

### 2.3.2. Experimental Design

To evaluate the effect of applying different nanofertilizers (Nf) to corn, two factors were considered: The first factor was the Nf, which was used as a fertilizer supplement according to four levels (potassium citrate nanoparticle solution [Nf1], natural zeolite powder [Nf2], zeolite impregnated with potassium citrate nanoparticle solution [Nf3], and filtered remnant solution [Nf4]); these materials were applied to each plant, where each plant was considered to be in 20 kg of soil (Table 3). The second factor was the application time in days after emergence (dae), with two levels (20 dae at vegetative stage V4 [T1] and 40 dae at stage V9 [T2]). These application dates correspond to the number of times nitrogen fertilizer was applied compared to conventional fertilization in the region; without mixing the nitrogen fertilizer and Nf. Additionally, a control (which received only conventional fertilization) was implemented for comparison purposes. A total of nine treatments were evaluated (Table 3).

**Table 3.** Nanofertilizer treatments and dosage per plant.

No.	Tmt <sup>†</sup>	Time (dae)	Nanofertilizer	Dose per plant	K (mg kg <sup>-1</sup> )	K <sup>+</sup> (cmol(+) kg <sup>-1</sup> )
0	Control	-	-	-	-	-
1	Nf1T1	20	Potassium citrate	13.00 ml	5.34	0.0137
2	Nf2T1	20	Natural zeolite	30.00 gr	6.75	0.0173
3	Nf3T1	20	Zeolite impregnated with potassium citrate	3.00 g	5.50	0.0141
4	Nf4T1	20	Filtering the remaining solution	13.00 ml	6.28	0.0161
5	Nf1T2	40	Potassium citrate	13.00 ml	5.34	0.0137
6	Nf2T2	40	Natural zeolite	30.00 gr	6.75	0.0173
7	Nf3T2	40	Zeolite impregnated with potassium citrate	3.00 g	5.50	0.0141
8	Nf4T2	40	Filtering the remaining solution	13.00 ml	6.28	0.0161

<sup>†</sup>Tmt: treatment; dae: days after emergence.

Each treatment had three replicates, resulting in 27 experimental units (EU). An EU consisted of a plot (2.5 × 10 m) with three furrows, and the usable plot measured one meter from the central furrow located in the center of the EU, which had six plants per meter. The experiment was conducted over two consecutive growing seasons on a plot of land with two contrasting soils [27] and a homogeneous spatial distribution. Given the two contrasting soil conditions (blocks) in the plot, the experiment employed a random complete block design with a factorial arrangement (4 × 2) and a control, with three replicates, over two crop cycles (two years).

### 2.3.3. Study Variables

Soil and plant variables were evaluated. The samples used for measurements were collected between 9:00 and 11:00 a.m.

The soil variables mainly consisted of chemical characteristics and were determined, in accordance with Mexican Official Standard NOM-021-REACT-2000 [31], as exchangeable cations (Ca<sup>2+</sup>, Mg<sup>2+</sup>, Na<sup>+</sup>, and K<sup>+</sup>) [ammonium acetate pH 7, 1 N] and soluble cations (saturated paste extract). The pH and EC were also measured from the saturated paste extract using a Hanna Instrument Model Hi-2211 potentiometer and conductivity meter. These variables were measured at the beginning and end of the experiment.

Measurements in plant were taken at 90 dae. In the field, the indirect chlorophyll content was obtained in SPAD (Soil and Plant Analysis Development) units on the leaf opposite the corn cob [32], using a Minolta® SPAD-502 chlorophyll meter. In addition, plant petioles were collected and placed in plastic bags, which were then stored in coolers with ice to keep the plant material fresh during transport to the laboratory. In the laboratory, the petiole extract was obtained [33], and the nitrate and potassium contents of the extracts were determined [34,35] using a Laquatwin® NO<sub>3</sub>-11 portable device and a K<sup>+</sup> model, respectively. All field and laboratory measurements were performed in triplicate.

At the end of the experiment, during field harvesting, the fresh weight (g), stem diameter (cm), and plant height (m) were measured; in the laboratory, the root dry weight (g) and plant dry weight (g) were registered, as well as the grain yield per ear (g).

## 2.4. Data Analysis

The results were subjected to analysis of variance ( $\alpha = 0.05$ ) using a linear mixed model. For this analysis, the treatments (factorial and control) and year (precipitation) were considered fixed-effect factors, and the blocks (B1 [Regosols] and B2 [Vertisols]) were considered random-effect factors. This allowed for the significance of the main effects and their interactions to be evaluated. Furthermore, it allowed for the evaluation of the treatment-year interaction, as well as the capture of soil variability, enabling inferences to be made to a broader soil population. This analysis was performed using the PROC MIXED procedure. Treatment and control means were compared using Dunnett's test ( $\alpha = 0.05$ ), while treatments with significant differences were compared using Tukey's test ( $p \leq 0.05$ ). These analyses were performed using the statistical package SAS/STAT 15.3 [36].

## 3. Results

### 3.1. Characteristics of the Materials Used as Supplements to Fertilization

The materials used as Nf had different morphological and chemical characteristics. Nf1 (solution with potassium citrate particles) consisted of spheroidal particles with an average diameter of 62.90 nm (Figure 2a) that occasionally appeared as accumulations of particles forming clumps, with a potassium concentration of 57.51% (Table 4). Our results support its use in the impregnation of zeolites. Furthermore, the zeta potential of the nanoparticles in this solution was -3.57 mv.

**Table 4.** Elemental analysis of nanofertilizers.

Element	Nf1 <sup>†</sup> (%) p/v	Nf2 (%)	Nf3 (%)	Nf4 (%) p/v
Oxygen	42.05	51.20	52.32	34.06
Sodium	0.00	0.70	-	0.40
Magnesium	0.00	0.56	0.17	0.00
Aluminum	0.00	7.17	5.83	0.00
Silicon	0.00	33.92	27.92	0.00
Potassium	57.51	3.05	12.35	46.63
Calcium	0.00	1.91	0.70	0.00
Iron	0.00	1.4	0.71	0.00

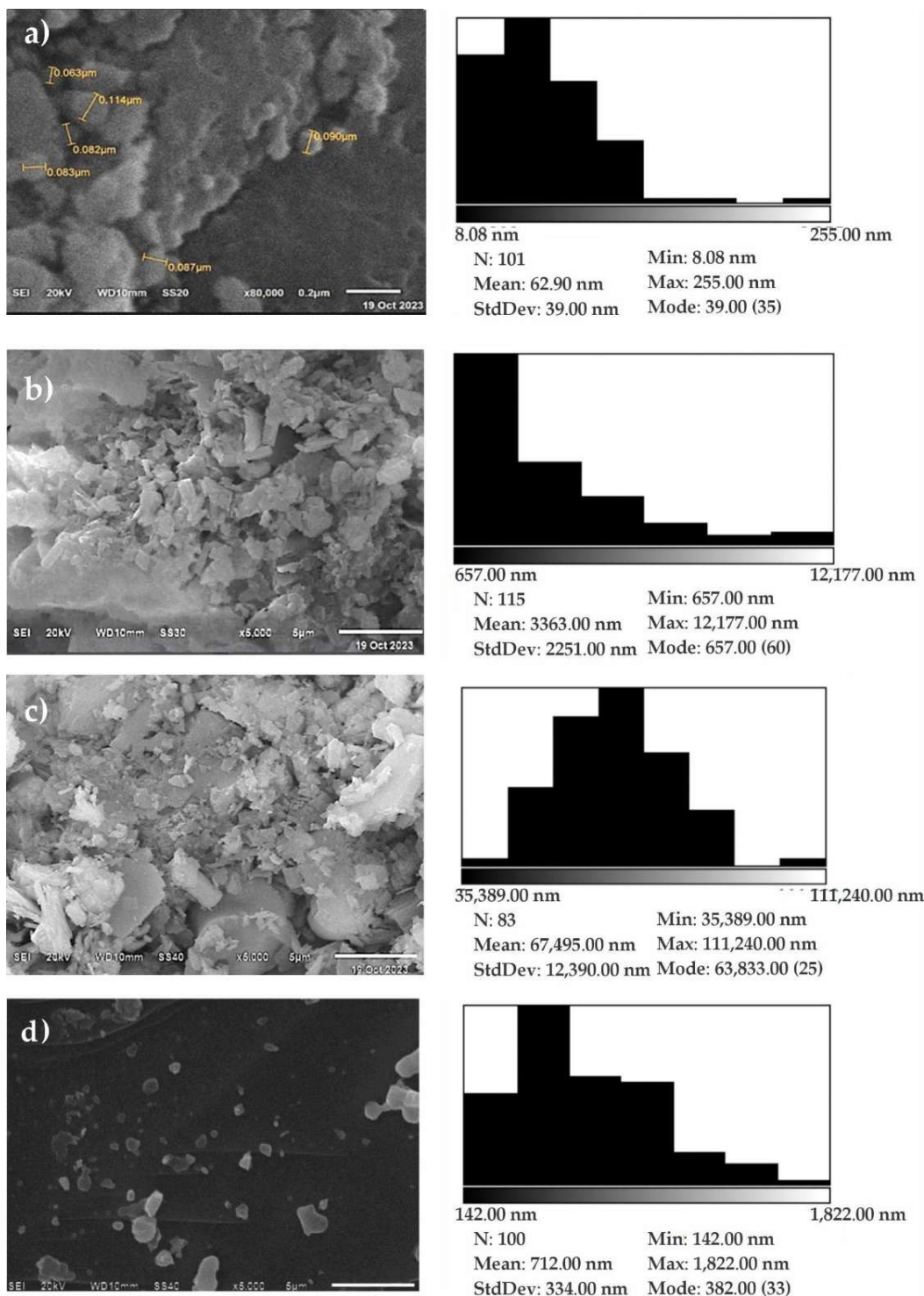
<sup>†</sup>Nf1: solution with potassium citrate particles; Nf2: natural zeolite 0.50 mm in diameter; Nf3: zeolite impregnated with potassium citrate; Nf4: filtrate of the supernatant solution.

In contrast, Nf2 (natural zeolite with a diameter of 0.25 mm) presented tabular and polyhedral granules (Figure 2b), with average diameters of 3360.00 nm and a potassium content of 3.05% (Table 4).

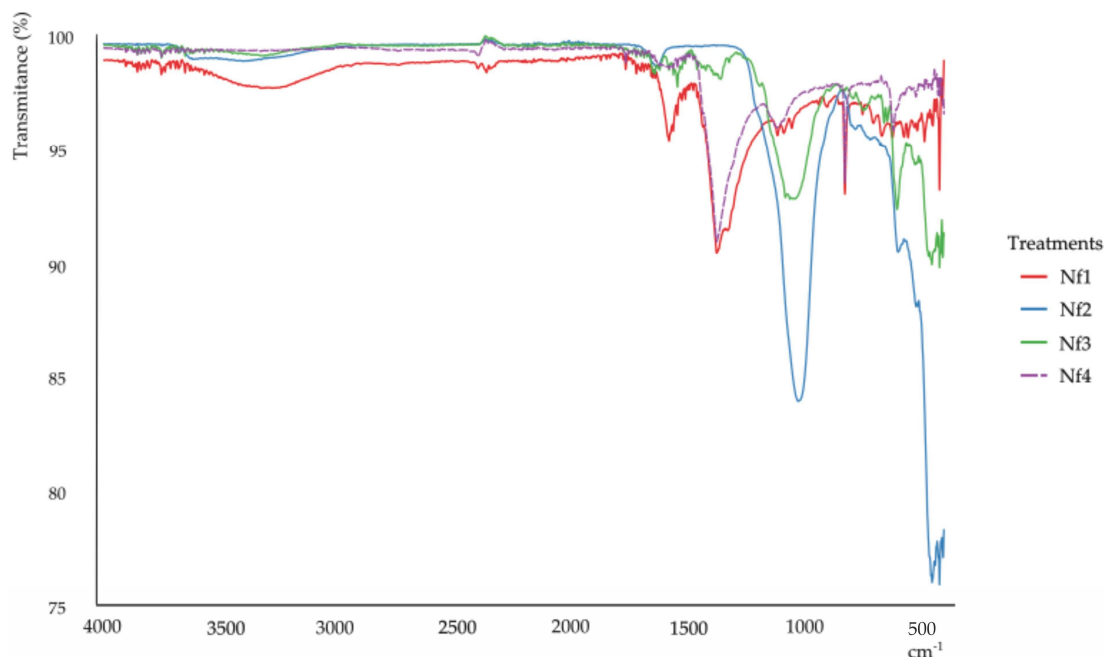
Likewise, zeolite impregnated with potassium citrate nanoparticle solution (Nf3) had tabular and polyhedral shapes with average sizes of 67,495.00 nm (Figure 2c) and a potassium concentration by weight of 12.35%. Note that accumulations of clumps and spherical particles were observed on the surface of the particles; in this case, the accumulation of potassium in Nf3 represents a 75% increase in potassium in the zeolite particles compared to Nf2. In contrast, the amount of potassium in Nf4 (remnant from the potassium citrate solution after impregnation) was 46.63%, with angular particles and an average diameter of 712.00 nm (Figure 2d).

Furthermore, the FTIR spectrum of Nf1 (potassium citrate) showed a broad absorption band centered at  $\sim 3400$   $\text{cm}^{-1}$ , attributable to the O-H stretching of the hydroxyl groups, as well as pronounced absorption peaks, among which those at 1572, 1372, 824, and 418  $\text{cm}^{-1}$  stood out (Figure 3). There were also small absorption peaks at 1680 and 1454  $\text{cm}^{-1}$ , which could correspond to the

asymmetric and symmetric vibrations of the carboxyl group characteristic of organic potassium salts. In contrast, the FTIR spectrum of Nf2 showed maximum absorption at  $1022\text{ cm}^{-1}$ , as well as stretching in the frequency band ranging from  $822$  to  $400\text{ cm}^{-1}$ , with absorption peaks detected at  $601$  and  $467\text{ cm}^{-1}$ . These data demonstrate the clear difference between Nf1 and Nf2, which was expected, since Nf1 consists of potassium citrate nanoparticles and Nf2 consists of clinoptilolite zeolite particles.



**Figure 2.** SEM image and particle size histograms of the nanofertilizers used: a) Nf1 (potassium citrate nanoparticle solution), b) Nf2 (natural zeolite powder) and c) Nf3 (zeolite impregnated with potassium citrate) and d) Nf4 (filtrate of the remaining solution from the impregnation process).



**Figure 3.** FTIR spectrophotometry of Nf1 (potassium citrate nanoparticles), Nf2 (natural zeolite), Nf3 (zeolite impregnated with potassium citrate) and Nf4 (remaining impregnation solution).

The Nf3 spectrum showed asymmetric stretching of lower intensity compared to Nf1 and Nf2. However, some absorption peaks found in Nf3 were located in absorption bands of both Nf1 and Nf2. For example, in the 1780–1630  $\text{cm}^{-1}$  band, an asymmetric absorption peak was detected at 1638  $\text{cm}^{-1}$ , as well as another peak at 1456  $\text{cm}^{-1}$  in the band between 1596 and 1426  $\text{cm}^{-1}$ . A broad band was observed between frequencies 1178 and 942  $\text{cm}^{-1}$ , with maximum absorption at 1024  $\text{cm}^{-1}$ , and a series of asymmetric stretches in the band from 822 to 400  $\text{cm}^{-1}$ , with absorption peaks at frequencies of 601 and 467  $\text{cm}^{-1}$ . These similarities in frequencies with Nf1 and Nf2 may be due to the Nf3 treatment involving clinoptilolite zeolite particles impregnated with potassium citrate solution, where interaction may occur between the particles of the materials that compose Nf3. The FTIR spectrum of Nf4 was like that of Nf1, although the absorption peaks were mostly weaker, which may be due to the composition of Nf4, as it was the residue of the impregnation solution of the zeolites with the potassium citrate nanoparticle solution.

### 3.2. Influence of Nanofertilizers on Soil and Plants

The variables evaluated in soil and plants showed differences ( $\alpha = 0.05$ ) according to the analysis of variance results (Tables 5 and 6). Note that the comparison of the factorial design against the control was significant in most cases, suggesting that the treatments had a different effect compared to the control. Furthermore, the Nf  $\times$  T interaction was not significant in some cases, indicating that the effect of the Nf treatments is independent of the application times. The year influenced all variables, which explains the influence of precipitation on the physiological processes of the plants. However, the Tmt  $\times$  Y interaction was not significant for all variables in the study, indicating that the treatment effects are consistent regardless of the precipitation levels over the study period. The blocks had an effect primarily on the soil variables (Table 5).

**Table 5.** *F* and *P* statistics of analysis of variance ( $\alpha = 0.05$ ) of edaphic variables.

Variable	Factor	gl	pH		EC <sup>†</sup>		K <sub>ex</sub>		K <sub>av</sub>	
			F <sup>‡</sup>	P <sup>‡</sup>	F	P	F	P	F	P
Treatment (Tmt)		8	6.16	***	1.25	ns	5.52	***	4.39	***
Control vs Treatments		1	3.58	*	0.35	ns	8.44	***	3.25	*
Factor Nf		3	5.48	***	2.88	*	6.73	***	7.27	***
Factor T		1	0.44	ns	3.56	ns	7.54	**	3.20	ns
Interaction Nf × T		3	0.53	ns	0.42	ns	0.38	ns	0.27	ns
Year (Y)		1	112.09	***	110.50	**	16.56	**	10.44	**
Interaction Tmt × Y		8	3.22	ns	0.55	ns	2.88	*	6.97	*
Block		1	8.42	*	85.30	**	50.18	**	12.45	**

<sup>†</sup>EC: electric conductivity; K<sub>ex</sub>: exchangeable potassium; K<sub>av</sub>: available potassium; *F*: *F* statistic; Nf: nanofertilizer; T: application time. <sup>‡</sup>*P*: significance (\* =  $P < 0.05$ ; \*\* =  $P < 0.01$ ; \*\*\* =  $P < 0.001$ ; ns = not significant).

**Table 6.** *F* and *P* statistics analysis of variance ( $\alpha = 0.05$ ) of plant variables.

Variable	Factor	gl	N-NO <sub>3</sub>		K <sup>†</sup>		CL		PH	
			F	P <sup>‡</sup>	F	P	F	P	F	P
Treatment (Tmt)		8	6.30	***	3.42	***	12.15	***	2.37	*
Control vs Factorial		1	3.21	*	2.73	*	4.22	*	3.29	*
Factor Nf		3	3.48	*	4.83	*	4.63	**	10.26	**
Factor T		1	0.87	ns	2.25	*	0.67	ns	5.36	*
Interaction Nf × T		3	1.24	ns	0.94	ns	0.85	ns	4.18	*
Year (Y)		1	2.45	***	175.86	***	48.32	***	10.40	**
Interaction Tmt × Y		8	4.37	*	0.54	ns	9.12	***	2.45	*
Block		1	10.52	***	2.07	ns	2.42	ns	1.45	ns

	gl	RDW		SD		PDW		YI	
		F	P <sup>‡</sup>	F	P	F	P	F	P
Treatment (Tmt)	8	9.56	***	2.31	*	11.34	***	5.31	***
Control vs Treatments	1	4.56	**	2.82	*	7.63	**	42.18	***
Factor Nf	3	10.45	**	3.49	**	9.38	***	4.56	**
Factor T	1	1.39	ns	4.82	*	2.74	*	3.42	*
Interaction Nf × T	3	2.30	ns	3.26	*	1.85	ns	2.28	*
Year (Y)	1	11.95	**	12.55	***	11.95	**	166.96	***
Interaction Tmt × Y	8	2.40	*	2.30	ns	6.15	*	2.72	*
Block	1	1.43	ns	1.18	ns	2.85	*	2.46	ns

<sup>†</sup>K: potassium in sap; N-NO<sub>3</sub>: nitrates in sap; CL: chlorophyll; PH: plant height; SD: stem diameter; PDW: plant dry weight; RDW: root dry weight; YI: yield; *F*: *F* statistic; Nf: nanofertilizer; T: application time. <sup>‡</sup>*P*: significance (\* =  $P < 0.05$ ; \*\* =  $P < 0.01$ ; \*\*\* =  $P < 0.001$ ; ns = not significant).

### 3.2.1. Effect of Nanofertilizers on Soil Variables

pH showed differences between treatments ( $\alpha = 0.05$ ) (Table 7). The Nf3T2 treatment caused a slight increase in pH from strongly acidic (5.10 in the control) to moderately acidic (5.63), while the smallest increase occurred with Nf1T1, maintaining a strongly acidic pH (5.05). However, pH behavior varied depending on the block (Table 7), with the trend remaining consistent in both years (Figure 4a). For example, the pH of B1 (Regosols) was slightly acidic (year1 = 5.46; year2 = 5.49), while

in B2 (Vertisols), it was strongly acidic (year1 = 4.89; year2 = 4.85); therefore, the nanofertilizer has influence on the soil pH (Figure 4a). Similarly, the timing of Nf application also influenced pH, as application at 40 dae resulted in a slightly acidic soil pH (Figure 4a) after harvest in years 1 and 2 (5.31 and 5.21, respectively), in contrast to application at 20 dae with strongly acidic pH.

**Table 7.** Average soil variables as a function of treatment, block and year.

No.	Treatment	pH	EC <sup>†</sup> (dS m <sup>-1</sup> )	K <sub>ex</sub> (cmol <sub>(+)</sub> kg <sup>-1</sup> )	K <sub>av</sub> (mg L <sup>-1</sup> )
0	Control	5.10 c <sup>‡</sup>	0.35	0.25 f	12.10 d
1	Nf1T1	5.05 c	0.31	0.42 d	13.50 c
2	Nf2T1	5.61 a	0.38	0.38 de	15.40 bc
3	Nf3T1	5.35 a	0.28	0.72 ab	18.20 b
4	Nf4T1	5.17 bc	0.40	0.51 c	16.10 b
5	Nf1T2	5.33 ab	0.36	0.39 de	14.20 bc
6	Nf2T2	5.61 a	0.45	0.64 b	14.80 bc
7	Nf3T2	<b>5.63 a</b>	0.42	<b>0.78 a</b>	<b>22.50 a</b>
8	Nf4T2	5.18 bc	0.42	0.48 cd	15.00 c
	<i>P</i>	**	ns	***	**
	HSD	0.26	-	0.06	2.45
<b>Block</b>					
	B1	5.02 b	0.45 a	0.12 b	14.20 b
	B2	5.35 a	0.22 b	0.41 a	28.69 a
	<i>P</i>	***	**	***	**
	HSD	0.15	0.08	0.04	1.80
<b>Year</b>					
	year1	4.88	0.52	0.27 b	10.40 b
	year2	5.18	0.21	0.47 a	35.80 a
	<i>P</i>	ns	ns	***	**
	HSD	-	-	0.03	1.55

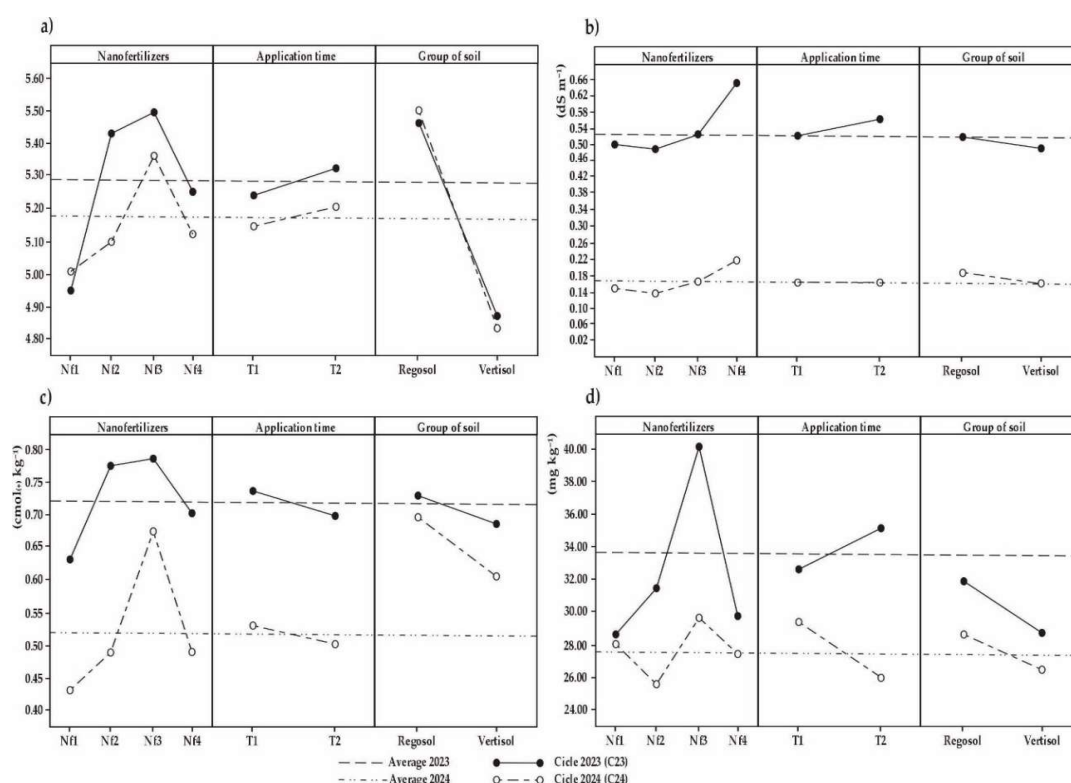
<sup>†</sup>EC: electric conductivity; K<sub>ex</sub>: exchangeable potassium; K<sub>av</sub>: available potassium; B1: Regosols; B2: Vertisols; year1: crop year 2023; S24: crop year 2024; HSD: honest significant difference. *P*: significance (\* =  $P < 0.05$ ; \*\* =  $P < 0.01$ ; \*\*\* =  $P < 0.001$ ). <sup>‡</sup>Different letters in the same column indicate significant differences (Tukey,  $p \leq 0.05$ ).

Nf also influenced EC; however, the values of this variable were less than 1.00 dS m<sup>-1</sup>. The Nf2T2 treatment caused an increase of 0.10 dS m<sup>-1</sup> in EC, reaching 0.45 dS m<sup>-1</sup> compared to the control (0.35 dS m<sup>-1</sup>). Therefore, the treatments did not promote soil salinity during the study period.

In contrast, the K<sub>ex</sub> in all treatments with nanofertilizers exceeded 0.38 cmol<sub>(+)</sub> kg<sup>-1</sup> with differences between treatments ( $\alpha = 0.05$ ). The highest and lowest concentrations were found in Nf3T2

(0.78  $\text{cmol}^{(+)} \text{kg}^{-1}$ ) and Nf2T1 (0.38  $\text{cmol}^{(+)} \text{kg}^{-1}$ ), respectively, which, compared to the control, represents increases of 212.12% and 52.12%, respectively (Table 7). The highest concentrations were found in Nf3T2 and Nf3T1 (0.78 and 0.72  $\text{cmol}^{(+)} \text{kg}^{-1}$ , respectively). This increase was greater than 100% in the concentration of  $K_{\text{ex}}$  compared to the control. In this regard, the effect of Nf3 is evident, as it had the greatest influence on all parameters compared to the other nanofertilizers (Figure 4c), while the lowest concentration was found in Nf2T1 (0.38  $\text{cmol}^{(+)} \text{kg}^{-1}$ ). In this sense, for this variable, the application date of the treatment influenced the  $K_{\text{ex}}$ . Thus, the application of Nf3 at 20 dae had a greater effect on  $K_{\text{ex}}$  (Figure 4c). Furthermore, only the Tmt  $\times$  Y interaction had a significant effect ( $F = 2.88$ ) on  $K_{\text{ex}}$  behavior (Table 5). Overall,  $K_{\text{ex}}$  concentration increased in Nf treatments compared to control concentration in each of the years evaluated.

The treatments showed differences in  $K_{\text{av}}$  concentration (Table 7). The highest  $K_{\text{av}}$  concentration was found in treatment Nf3T2 (22.50  $\text{mg L}^{-1}$ ).  $K_{\text{av}}$  content increased from 11.57% to 85.95% compared to the control (12.10  $\text{mg L}^{-1}$ ). Similarly, the blocks (B1 = 14.20  $\text{mg L}^{-1}$ ; B2 = 28.69  $\text{mg L}^{-1}$ ) and years (year1 = 10.40  $\text{mg L}^{-1}$ ; year2 = 35.80  $\text{mg L}^{-1}$ ) influenced  $K_{\text{av}}$ .

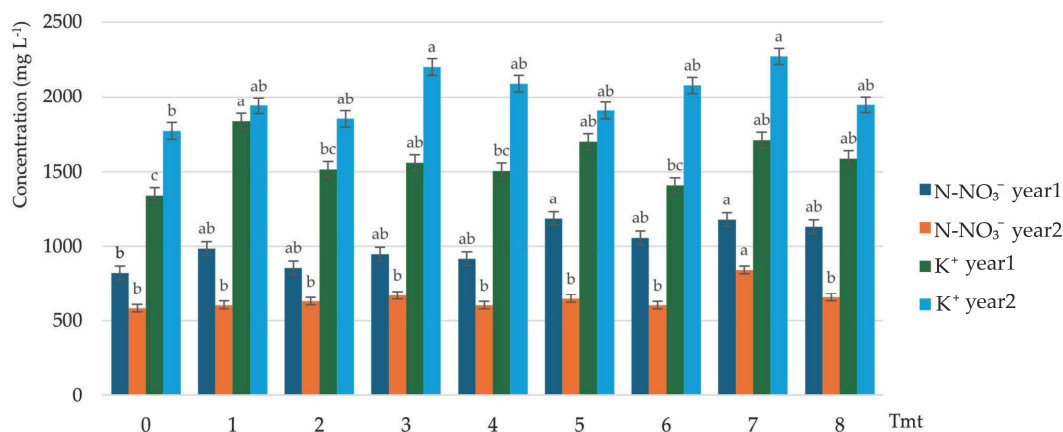


**Figure 4.** Main effects of the study factors on soil variables: a) pH; b) electrical conductivity (EC); c) exchangeable potassium; d) available potassium.

### 3.2.2. Effect of Nanofertilizers on the Plant Variables

Treatment, block, and year influenced the behavior of the study variables in the plant ( $\alpha = 0.05$ ). For petiole sap, the highest concentrations of nitrates (1180.00 and 843.00  $\text{mg L}^{-1}$ ) and potassium (1836.00 and 2270.00  $\text{mg L}^{-1}$ ) in years 1 and 2 were obtained with treatment Nf3T2 (Figure 5).

The differential behavior of nitrates and potassium in the cultivation cycles is notable, since the nitrate concentration in the sap decreased from year1 to year2, while the potassium concentration increased. This was evident in the case of nitrates, where significant interactions were observed (Tmt  $\times$  Y [ $F = 4.37$ ]); in contrast, this interaction had no effect on potassium in the sap (Table 5). This differential behavior of nitrates and potassium in relation to the treatments can be attributed to the climatic conditions that prevailed during each year of the study, mainly precipitation, given that the crop was rainfed.



**Figure 5.** Effect of treatments on nitrate and potassium content in sap of maize plants from two growing cycles (2023 and 2024).

Conversely, treatment, block, and year also influenced ( $\alpha = 0.05$ ) plant height (PH), root dry weight (RDW), stem diameter (SD), and plant dry weight (PDW). The variables showed differences between treatments (Tukey,  $p \leq 0.05$ ) (Table 8). For example, the greatest PH was obtained with the Nf3T2 treatment (2.68 m); while the shortest plant was found in the control (2.42 m).

**Table 8.** Average values of plant variables as a function of treatment, block and year.

No.	Treatment	CL <sup>†</sup> (SPAD units)	RDW (g)	SD (mm)	PH (m)	PDW (g)
0	Control	54.40	53.63 b <sup>†</sup>	15.70 d	2.42 c	102.10 f
1	Nf1T1	54.10	68.61 ab	17.50 c	2.55 b	115.80 de
2	Nf2T1	54.60	68.20 ab	16.80 cd	2.66 a	108.40 ef
3	Nf3T1	54.90	83.19 a	20.20 a	2.61 ab	134.60 bc
4	Nf4T1	55.30	60.67 b	19.80 ab	2.50 bc	138.20 b
5	Nf1T2	55.30	69.83 ab	18.40 bc	2.66 a	128.90 c
6	Nf2T2	55.20	70.62 ab	17.90 c	2.65 a	142.50 b
7	Nf3T2	54.60	<b>81.82 a</b>	<b>21.50 a</b>	<b>2.68 a</b>	<b>156.40 a</b>
8	Nf4T2	54.10	53.11 b	16.50 cd	2.64 a	110.30 e
	<i>P</i>	ns	***	**	**	**
	HSD	-	14.21	1.97	0.08	11.40
<b>Block</b>						
	B1	52.10 b	64.25	16.80 b	2.53	115.40 b
	B2	57.20 a	68.99	19.70 a	2.74	129.50 a
	<i>P</i>	**	ns	*	ns	**
	HSD	1.49	-	0.90	-	7.50
<b>Year</b>						
	year1	55.40 a	55.90 b	18.50	2.58	124.10
	year2	53.90 b	77.25 a	18.00	2.58	120.80
	<i>P</i>	**	***	ns	ns	ns
	HSD	1.10	7.56	-	-	-

<sup>†</sup>CL: chlorophyll; RDW: root dry weight; SD: stem diameter; PH: plant height; PDW: plant dry weight; B1: Regosols; B2: Vertisols; year1: crop year 2023; S24: crop year 2024; HSD: honest significant difference; *P*:

significance (\* =  $P < 0.05$ ; \*\* =  $P < 0.01$ ; \*\*\* =  $P < 0.001$ ). †Different letters in the same column indicate significant differences (Tukey,  $p \leq 0.05$ ).

Meanwhile, the RDW in the Nf3T2 treatment showed the highest weight (81.82 g). In the case of SD, differences were also found between treatments, where Nf3T2 again showed the largest diameter (21.50 mm). PDW differed between treatments, with Nf3T2 plants of higher dry weight (156.40 g). Note that the treatments were always superior to the controls with regard to the variables studied. It is important to highlight that the block and year effects on some variables were not significant, indicating that the treatment effects were independent.

Similarly, the treatments also influenced corn grain yield. In this regard, Nf3T2 had the highest yield in year1 and year2 (151.80 and 216.90 g, respectively), while the lowest yield (77.30 and 140.60 g) was recorded in the control in both years (Figure 6). Once again, this demonstrates the influence of the higher potassium content found in this treatment and the effect of the weather conditions that existed in each year of the study. The effect of applying zeolite with potassium nanoparticles 40 days after sowing caused a 96.30% increase in the year 2023 compared to the control, while in the year 2024, there was a 47.90% increase compared to the control for that year, which is related to the availability of potassium and water during each crop cycle, as seen in the significant effect of the Tmt  $\times$  Y interaction ( $F = 2.72$ ) (Table 6).

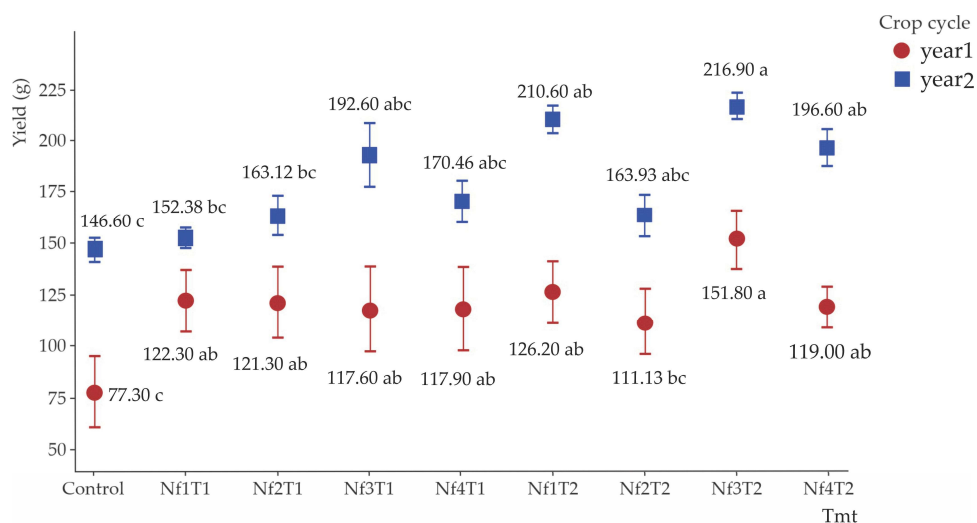


Figure 6. Effect of treatments on grain yield per maize plant in the 2023 and 2024 growing seasons.

## 4. Discussion

### 4.1. Characteristics of Materials

Nanomaterials are particles ranging in size from 1 to 100 nm [37,38], and nanoparticles containing nutrients such as N, P, and K in their structure are considered nanofertilizers [39]. Furthermore, the spheroidal shape of nanoparticles is a distinctive feature of their nanometric size in all three dimensions, although they can also be cubic, tabular, or prismatic, but the size range of the nanomaterial must be upheld [40]. These morphological characteristics are similar to those presented by the Nf1 in our experiment. However, the potassium citrate nanoparticles in our study are unstable in solution due to their zeta potential (-3.7 mV) [41]; these nanoparticles would clump together in the solution over time [42]. Nevertheless, this did not pose a problem in our study, since the solution was first used to impregnate the zeolite particles and then used as a treatment, applied directly to the soil, regardless of whether the particles were present as nanoparticles or larger clumps, as observed in our study. Moreover, the FTIR spectrum of potassium citrate exhibited different absorption bands [43]. The first absorption band was broad (3400–3000  $\text{cm}^{-1}$ ) due to the vibration of the O-H bond [44,45].

However, absorption can sometimes be reduced by vibrations of other bonds in the molecule, such as through C=O and C–O bonds with vibrations between 1769 and 1680  $\text{cm}^{-1}$  and 1250 and 1100  $\text{cm}^{-1}$ , respectively [44,46]; the vibration of the K–O bond, which can be detected from 1400 to 1372  $\text{cm}^{-1}$  and is characteristic of potassium nanofertilizers [47]; and the curvature produced by C–H bonds at a frequency of 1485 to 1445  $\text{cm}^{-1}$  [48]. This allows us to conclude that the potassium citrate nanoparticles synthesized in our research constitute potassium citrate nanofertilizer. Regarding its morphology, the clinoptilolite zeolite studied via SEM is a mineral with defined crystals that can vary in size, in the form of sheets or prismatic granules that can form larger structures in the form of aggregates, whose sizes vary from micrometers to nanometers [49,50], with a rough surface and the presence of mesopores and micropores [51]. The FTIR spectrum of zeolite is commonly associated with vibrations around the frequencies 1050, 790, and 450  $\text{cm}^{-1}$ , characteristic bands of clinoptilolite zeolite [52]. Other authors have reported an absorption peak at  $\sim 1022$   $\text{cm}^{-1}$ , caused by the vibration of Si–O bonds [53,54]. However, Khanna [55] indicated that O–Si–O bonds were detected at 463  $\text{cm}^{-1}$ . Similarly, the vibration of the K–O bond has been reported in an absorption peak between 824 and 463  $\text{cm}^{-1}$  [56]. In this regard, Díaz-Nava et al. [57] indicated that the displacement of absorption peaks tends to be due to the presence of cations in the exchange complex of zeolites and the origin of the mineral, without a change in its crystalline structure. The spectrum of materials impregnated with smaller particles of different types tends to show spectral characteristics of both materials but is attenuated [58]. Zeolites have pores or empty spaces of different sizes (from mesopores to nanopores), which is why they are considered nanoconfiners and act as reactors, where compounds with metal ions can crystallize [18]. In this regard, it is likely that when the impregnation solution penetrates into the pores, after drying, some potassium citrate nanoparticles crystallize inside the pores and others do so on the surface of the outer part of the zeolite particles. However, information related to this aspect is scarce. If the above is proven, it could explain why the morphological characteristics of the product of impregnating zeolites with potassium citrate nanoparticles are similar to those of clinoptilolite zeolite, but with the FTIR spectrum showing attenuated peaks compared to the spectra of the initial materials of these particles due to the probable interference caused by the functional groups (carboxyls) of potassium citrate, as well as the potassium–oxygen or silicon–oxygen bonds of clinoptilolite zeolite in the potassium citrate-impregnated zeolite particles. This means that the zeolite particles are impregnated with potassium citrate nanoparticles, which is significant from an agricultural perspective, since zeolites impregnated with potassium citrate represent a potassium reserve in the soil that cannot be easily lost through percolation processes; instead, depending on the moisture or condition of the soil solution, the potassium would become available to plants, as explained below.

#### 4.2. Influence of Nanofertilizers on Soils

The slight increase in soil pH after adding clinoptilolite zeolite has been reported by other researchers [59,60]. This effect stems from zeolite's alkaline nature, which helps neutralize the pH of acidic soils [61,62]. In this regard, Nur Aainaa et al. [63] indicated that in soils with a pH lower than 5.5, reactivity is governed by aluminum hydrolysis rather than by the exchange of  $\text{H}^+$  ions for cations found at the exchange sites of the zeolite, such that pH variations are dampened. This means that the potassium citrate-impregnated clinoptilolite zeolite in our study could have two effects: increasing the pH and supplying potassium to the soil. Firstly, there is a slight increase in pH due to the decrease in  $\text{Al}^+$  [64], coupled with the exchange of  $\text{H}^+$  ions in the soil solution for the  $\text{K}^+$  ions found in the zeolite exchange sites [65–67]. Secondly, the contribution of  $\text{K}^+$  that was part of the potassium citrate to the soil solution since potassium citrate can dissolve rapidly in the soil solution, releasing potassium ions that are readily available to plants [68]. According to Wang et al. [69], the easy release of available potassium increases when exchangeable potassium increases. However, naturally available potassium depends on the type of primary mineral containing the potassium, the type of clay that can fix it, and the exchangeable potassium and availability of exchange sites [60]. However, environmental factors (such as precipitation) and anthropogenic factors (irrigation) also influence it,

as they can promote the leaching of potassium ions. Potassium fertilizers can be lightly and frequently applied to reduce leaching losses, decreasing the chances of fixation in unavailable forms [70]. This explains the increase in available and exchangeable potassium in the soils after each crop cycle in our study. Despite this, the presence of available potassium at the end of harvest suggests that potassium citrate-impregnated clinoptilolite zeolite could slowly release potassium into the soil solution. This relates to the potassium release mechanisms in these impregnated particles, where the timing of application does not influence the process, as the particles continue to release potassium over time, making it available and varying according to the soil and its moisture (influenced by precipitation or irrigation).

#### 4.3. Effect of Nanofertilizers on the Plants

Applying potassium to the soil improves the plant's absorption of nitrogen and potassium, optimizes nitrogen metabolism within the plant, and promotes photosynthesis and the transport of photosynthates [71,72]. One way to verify the nitrogen and potassium content in the plant is to evaluate the sap in the petiole, which reflects the nutritional status of the plants [73]. In this regard, the relationship between  $K^+$  and  $NO_3^+$  contents in corn sap in our study was similar to that reported in other studies with different plants [74]. The increase in potassium and nitrogen in the form of nitrates translates into an increase in the yield components of corn, since potassium participates in various functions within plants such as enzyme activation, the maintenance of the electrical potential of the cell membrane, cell expansion, and osmoregulation, and even confers responsiveness to abiotic stress [75]. This is reflected in the plants in our study, since the presence of readily available potassium as a result of the application of zeolite impregnated with potassium citrate to the soils studied led to an increase in plant height, dry matter (plant and root), stem diameter, and grain yield in corn plants. Similar results from the application of potassium to corn have been reported in other studies [76,77]. The effect of soil on plant response to potassium addition has been documented, establishing that soil texture class and mineralogy can influence potassium availability, which is reflected in the phenological characteristics of corn plants and their yield [70,78]. This explains the differential effect of potassium addition on plants due to soil type in our research. However, the response of yield variables to the addition of potassium may vary depending on the amount of water available during the growing cycle, especially in rainfed crops. In this regard, corn yield variables under drought conditions can be increased when potassium is added to the soil, even tending to increase as the volume of irrigation or the precipitation and the amount of potassium increase [79–81], since this element, in the presence of water within the plant, promotes plant development, cell division, and carbohydrate generation [82]. When potassium is added to the soil in installments, the plant's response favors increased yield [77], which was observed in our research. When zeolite impregnated with potassium citrate nanoparticles (20 or 40 dae) was added as a supplement to the initial fertilization, the corn grain yield was higher than that of plants that did not receive the supplement. Notably, the yield was better at 40 dae, with increases of more than 47.00%. In this sense, Singh et al. [83] indicated that since flowering and grain filling are critical stages for plants, yield increases when plants have readily available potassium during these stages [84]. This allows us to deduce that zeolite impregnated with potassium citrate nanoparticles, when added to the soil as a supplement to the conventional fertilizer dose, increases the concentration of  $K_{ex}$  and  $K_{av}$ , leading to an increase in the yield of corn plants. However, the behavior of this type of material must be studied under other conditions and with different crops to verify its effectiveness.

## 5. Conclusions

The addition of clinoptilolite zeolite particles impregnated with potassium citrate nanoparticles as a supplement to corn crop fertilization in acidic soils increases the concentration of potassium available to plants during the stages of highest crop demand when applied 40 days after emergence. This was demonstrated by obtaining a concentration of available potassium greater than  $20.00 \text{ mg L}^{-1}$  at the end of the harvest in two consecutive years. Clinoptilolite zeolite impregnated with potassium

citrate nanoparticles promotes a slight increase in pH, an effect that prevailed throughout the two continuous crop years until harvest. In this regard, this zeolite acted independently of the existing acidic soil conditions. Our findings leads us to assume that clinoptilolite zeolite impregnated with potassium citrate nanoparticles represents an alternative treatment for crop fertilization in acidic soils, if used as a complement to conventional fertilization. However, it is important to validate its effectiveness with other crops and in other soils, as well as to monitor it over time to determine the duration of the effect of this type of fertilizer supplement.

**Author Contributions:** O.A.-C. and M.Á.S.-C. participated in the conceptualization, methodology, data collection, data processing with software, formal analysis, project administration, supervision, writing—review and editing, and writing—original draft preparation. J.F.G.-L. and H.F.-M. participated in the formal analysis, review, and editing of the original draft preparation. N.A.M.-M. and H.S.-E. participated in the formal analysis and review. All authors have read and agreed to the published version of the manuscript.

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**Data Availability Statement:** The data presented in this study is available on request from the corresponding author. The data is not publicly available due to privacy restriction.

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**Conflicts of Interest:** The authors declare no conflicts of interest.

## Abbreviations

B1	Block one (Regosols)
B2	Block two (Vertisols)
CEC	Cationic exchange capacity
CL	Chlorophyll
dae	Days after of emergence
EC	Electric conductivity
EDS	Elemental analysis via X-ray dispersive spectrometry
EU	Experimental unit
FTIR	Fourier transform infrared spectroscopy
HSD	Honest significant difference
K <sub>av</sub>	Available potassium
K <sub>ex</sub>	Exchangeable potassium
Nf	Nanofertilizer
Nfs	Nanofertilizers
Nf1	Potassium citrate
Nf2	Natural zeolite
Nf3	Zeolite impregnated with potassium citrate
Nf4	Filtering the remaining solution
Nf1T1	Treatment one: Potassium citrate added 20 dae
Nf2T1	Treatment two: Natural zeolite added 20 dae
Nf3T1	Treatment tree: Zeolite impregnated with potassium citrate added 20 dae
Nf4T1	Treatment four: Filtering the remaining solution added 20 dae
Nf1T2	Treatment five: Potassium citrate added 40 dae
Nf2T2	Treatment six: Natural zeolite added 40 dae
Nf3T2	Treatment seven: Zeolite impregnated with potassium citrate added 40 dae
Nf4T2	Treatment eight: Filtering the remaining solution added 40 dae
Tmt × Y	Interaction between treatment and year
OM	Organic matter
PH	Plant height
PDW	Plant dry weight

RDW	Root dry weight
SAS	Statistical analysis system
SD	Stem diameter
SEM	Scanning electron microscopy
SPAD	Soil and plant analysis development
T	Application time
Tmt	Treatment
Y	Year
year1	Crop cycle 2023
year2	Crop cycle 2024
Yl	Yield

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