



Enhancing wheat yield (*Triticum durum* Desf.) through the application of TiO_2 , ZnO , Fe_xO_x , and Ag nanoparticles to agricultural land

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Abstract

As the world's population continues to grow, the demand for food is also rising. To meet this demand, the Food and Agriculture Organization (FAO) has proposed using nanoparticles (NPs) as a new tool for agriculture. Compared to traditional agrochemical fertilizers, NPs are more efficient and environmentally friendly. Studies have shown that NPs can improve soil quality and increase crop yields. To evaluate the effectiveness of four types of NPs— TiO_2 , ZnO , Fe_xO_x , and Ag—on the properties of a Samayoa C2004 wheat crop, a field experiment was conducted in southeastern Coahuila state, Mexico. The experiment involved planting a dose of 140 kg ha^{-1} in plots of 9 m^2 (divided by 6 m^2 borders), with a dose of NPs set at 200 g ha^{-1} . The data was analyzed using a one-way ANOVA and Tukey test ($p \leq 0.05$) in Minitab18 software. The study results showed that the ZnO -NPs treatment increased the yield compared to the values reported by Mexico's National Institute for Forestry, Agriculture and Livestock Research (INIFAP) in the same cycle. The soil biomass was also increased compared to the other treatments, which is attributed to the beneficial properties of the ZnO -NPs. In conclusion, this study found that ZnO -NPs were the most effective treatment for improving soil quality and increasing wheat yield under field conditions in semiarid soil.

Keywords Agricultural nanotechnology · Crop nutrition · Soil fertility · Semiarid soil · Soil quality

Introduction

Agriculture is a crucial primary activity that sustains eight billion people worldwide. However, the excessive use of chemical fertilizers has resulted in soil degradation and climate change, causing significant environmental harm. While nanotechnology has been employed in recent years to enhance crop production and safeguard crops, more research is required to determine the impact of nanomaterials (NMs) or nanoparticles (NPs) on soil properties and their relationship with soil microorganisms over an extended period (Gardea-Torresdey et al. 2014; Roco 2003).

There are several studies about the most used NPs around the world. For example, in agriculture, titanium dioxide NPs (TiO_2 -NPs) interact with soil humic acids and plant root exudates before moving through the plant roots. Besides increasing the enzymatic activity of chlorophyll and carotenoids, N_2 fixation, and Fe and Mg uptake (Cox et al. 2016; Landa 2021); Zinc oxide NPs (ZnO -NPs) provide plants with Zn micronutrients, promote growth, and act as antioxidants; these NPs help with abiotic stress like drought, salinity and UV radiation (Babaei et al. 2017); A mixture of iron oxide NPs containing Fe_2O_3 plus Fe_3O_4 (Fe_xO_x -NPs) help soil bacteria (plant growth promoting rhizobacteria: PGPR) grow without affecting microbial diversity, resulting in better plant growth and they can act as very important nutrient in a ion form (Rajput et al. 2020); and also, low doses of silver NPs (Ag-NPs; 10 ppm) are an antioxidant, leading to higher crop yields of ryegrass grass, barley, and flax, these NPs increase the uptake of water in seeds, catalase, chlorophyll, carotenoids, and bioavailability of K, Ca, and S (El-Temsah and Joner 2012; Landa 2021).

Introducing NPs in agriculture is a promising method to increase productivity and yield. Nano agrochemical

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fertilizers are more effective than traditional bulk agrochemical fertilizers. Consequently, nano agrochemical fertilizers must be tested in natural conditions (Dimkpa et al. 2019; Tian et al. 2019).

Wheat cereal, including *Triticum aestivum* L. and *Triticum durum* Desf, provides food for almost 28% of the world's population. Novel strategies must be implemented to meet the predicted 60% demand for wheat by 2050 (Ahmadian et al. 2021; Barreras-Urbina 2020). In the north and center of Coahuila state, wheat has been the most significant crop since 2017, according to Mexico's National Institute for Forestry, Agriculture and Livestock Research (INIFAP). In this study, we used Samayoa C2004 wheat, a semi-dwarf variety that yields 5.2 to 8.3 t ha⁻¹ (Camacho-Casas et al. 2010), with the aim of evaluating the effect of NPs (ZnO, TiO₂, Fe_xO_x, and Ag NPs) on wheat yield and the physicochemical properties of soil to determinate which NPs better improve the yields and plant characteristics in agricultural land conditions. This study hypothetical that using different NPs to fertilize *Triticum durum* Desf growing in agricultural soil under a semiarid land condition could significantly improve the yield and crop characteristics, considering that the used NPs in this research contain beneficial elements or crop nutrients.

Materials and methods

A study was conducted in field conditions in Hda. San Isidro de Gómez, General Cepeda, Coahuila de Zaragoza, Mexico (Latitude 25°18'05", Longitude -101°23'26") where agricultural soil (leptosol) samples were collected from a field with a depth of 0–20 cm. This field was previously used for corn crops without fertilizer using conventional farming practices at least ten years ago.

Durum wheat variety samayoa C2004 (*Triticum durum* Desf.)

Samayoa C2004 was a bred plant adapted for semiarid conditions and resistant to dry climates and shallow, nutrient-depleted soil. This semi-dwarf cultivar is resistant to leaf rust, and the quality characteristics of pasta-making are better than that of the commercial control cultivar Júpare C2001. Therefore, Samayoa C2004 becomes a better option for international export for wheat producers from northwestern Mexico. During crop seasons of evaluation 2001–2002 to 2003–2004, Samayoa showed an average grain yield of 6.7 t ha⁻¹. Samayoa C2004 is a semi-dwarf plant measuring 80 cm in height, with a vegetative cycle of 78 days to heading and 124 to physiological maturity. The plant has an erect growth habit, and the frequency of plants with curved flag leaves is absent or low. At maturity, the

stems are white with a thin pith in cross-section. The spike has a pyramidal profile, medium density, and is short (5.7 to 7 cm), excluding the awns or beards, which are longer than the spike.

NPs characterization

The NPs used were bought at 'Investigación y Desarrollo de Nanomateriales, S.A. de C.V.' The morphological characterization and chemical composition were determined by field emission scanning electron microscopy with energy dispersive spectroscopy (FESEM-EDS: JEOL JSM-7800F) with 10.0 kV acceleration voltage, and the crystal structure was determined by x-ray diffraction (XRD: Xpert-Philips PW3040 diffractometer).

Soil characterization

The normalized method was utilized to obtain twelve soil samples (Sosa and Alavarenga 2012). The soil samples were stored in refrigeration at 4 °C for physicochemical characterization and another soil sample at – 18 °C for soil microbial biomass determination. The soil samples were dried at greenhouse conditions for 72 h before characterization and were sieved at 2 mm (Pérez Moreno et al. 2019).

A potentiometer Orion Star A211 Thermo Scientific determined the pH level with a 1:2.5 soil-to-water ratio. The electrical conductivity (EC) (Pérez Moreno et al. 2019) was also determined using a conductometer and an Orion Star A212 Thermo Scientific. Soil organic matter (SOM) was measured using the calcination method (Sarabia-Castillo et al. 2023) using a muffle Thermolyne Thermo Scientific. The gravimetric soil humidity (Hg) was determined with a humidity analyzer OHAUS MB90, and the test tube method determined an apparent density (pa) (Salazar-Calvo et al. 2020). The texture was determined through the hydrometric method and USDA classification with Bouyoucus (ASTM 152-H). The microbial abundance (microbial soil biomass) was measured with the fumigation-extraction method and a UV–Vis Jenway 7305 spectrophotometer (Albiach et al. 2006), and the concentration of total K and total P was determined by inductively coupled plasma emission spectrometry (ICP-OES) using a Perkin Elmer Optima 8300 spectrometer (Sarabia-Castillo et al. 2023).

Field experiment and samplings

The experiment was designed with randomly divided plots measuring 9 m² by 6 m² borders, with five treatments and five replicates. The study included 25 experimental units that evaluated the effects of four NPs (TiO₂, ZnO, Fe_xO_x, and Ag) and a control treatment without NPs. Before sowing, the

plots were hydrated with a layer of 150 mm of water, then, in each plot, seeds of durum wheat variety Samayoa C2004 (*Triticum durum* Desf.) were sown with a sowing density of 140 kg ha^{-1} ; subsequently, NPs suspensions (in 500 mL of deionized water) at a concentration equivalent to 200 g ha^{-1} were prepared, subjected to an ultrasound bath (Autoscience As2060B). They added to each plot individually according to the established treatments.

The crop was irrigated according to its needs (800 mm of water per cycle); the experiment was maintained for 130 days; during this period, two destructive and random samplings were carried out at 60 and 130 days after sowing (DAS) using a visor device with a 10 cm diameter (Guzmán-Báez et al. 2021). Due to the total experiment being too big, the establishment of pseudo samples to represent all the fields, the pseudo sample size was determined using Eq. $n = (t^2 v, \alpha) / X(D)^2$ an error margin of 20% (D), a t-student value of 4, and a confidence $\alpha = 0.05$ (Gaillard de Benítez and Pece 2011).

In each sampling, the length of the aerial biomass of the wheat plants was measured; in the case of samples taken at 130 DAS, the length of the spikes was also measured. Subsequently, the samples were placed in paper bags and dried in a Thermo Scientific forced air oven at 72°C for 72 h or until constant weight to determine the dry biomass weight.

To compare crop yield, the harvest seeds at 130 DAS were weighed and compared with the annual average in the region under similar climate conditions to data obtained from Valley Yaqui INIFAP [11] during a fall-winter experiment.

Statistical analysis was conducted using a one-way analysis of variance (ANOVA), and means were compared using the Tukey test with a significance level of $p \leq 0.05$, utilizing Minitab 18 software.

Results

NPs characterization

The XRD analysis (2θ degrees) of TiO_2 -NPs revealed the presence of two crystalline phases that can be identified as the anatase phase with the planes (100), (004), (200), (211), (204), and (215), corresponding to card number 96–900–8215. The second phase found is the Brookite phase with the planes (121) and (233), corresponding to card number 96–900–4140. Besides, in the FESEM micrograph, the TiO_2 -NPs exhibited an irregular and heterogeneous morphology that tended to form clusters (Figs. 1a and b).

In XRD ZnO -NPs analysis, the planes (100), (002), (101), and (102) were identified in comparison with card number 00–901–1662. This indicates that they have

a hexagonal wurtzite phase structure. Regarding their morphology, elongated particles in the form of prismatic bars are observed in Figs. 1c and d.

The Fe_xO_x -NPs exhibit two crystalline phases (2θ degrees), the hematite phase, represented by the presence of the planes (42–2), (51–4), and (44–4), corresponding to card number 96–900–0140; and the magnetite phase with planes (-220), (222), and (440) from card number 96–900–2317. Regarding their morphology, they are spherical or semi-spherical particles with a high degree of aggregation (Fig. 1e and f).

The Ag -NPs exhibit four diffraction peaks (2θ degrees) (111), (200), (220), and (311) compared to card 01–089–3722, corresponding to the diffraction planes of silver with an FCC structure. It can be observed that the morphology of the Ag-NPs is irregular, with a high degree of aggregation (Figs. 1g and h).

Soil characterization

The initial properties of the soil were a pH of 7.1, electrical conductivity of 0.340 dS m^{-1} , soil organic matter of 4.2%, and apparent density of 1.2 g cm^{-3} . The soil texture was loamy clay with 43% sand, 29% silt, and 28% clay. The gravimetric humidity was 3.99%. Soil microbial biomass carbon was $126.13 \text{ mg kg}^{-1}$, and the principal total elements were: K was 2.11 g kg^{-1} , P was 0.2 mg kg^{-1} , N was 1.7 g kg^{-1} , C was 29.2 g kg^{-1} , and TOC 11.63 g kg^{-1} .

Field experiment

In Table 1 it is observed that at 60 DAS, the pH was lower in the TiO_2 treatment (7.4 ± 0.04), with the highest pH recorded in the ZnO (7.7 ± 0.02) and Fe_xO_x (7.7 ± 0.02) treatments, compared with control treatment (7.5 ± 0.01). No significant differences were observed between the treatments by the end of the harvest period (130 DAS) while it maintains values between 8.7 and 8.8. The soil electrical conductivity (EC) at 60 DAS was lower in ZnO treatment ($0.5 \pm 0.03 \text{ dS m}^{-1}$) compared with control treatment (0.6 ± 0.02). At 130 DAS no significant differences were observed and the values were between 0.1 and 0.3 dS m^{-1} (Table 1).

Soil organic matter did not show significant differences between the treatments at 60 DAS. At 130 DAS the soil with TiO_2 ($4.7 \pm 0.25\%$) and Ag ($5.1 \pm 0.27\%$) treatments showed a lower value compared with control treatment ($5.9 \pm 0.01\%$). The potassium (K) concentration at 60 DAS was lower in the Ag NPs treatment ($1.5 \pm 0.00 \text{ g kg}^{-1}$) compared to the control treatment ($2.4 \pm 0.01 \text{ g kg}^{-1}$). By 130 DAS, the highest total K concentration was observed in the Fe_xO_x ($3.6 \pm 0.02 \text{ g kg}^{-1}$) and Ag NPs treatment ($3.4 \pm 0.00 \text{ g kg}^{-1}$) compared to the control treatment ($2.9 \pm 0.00 \text{ g kg}^{-1}$; Table 1).

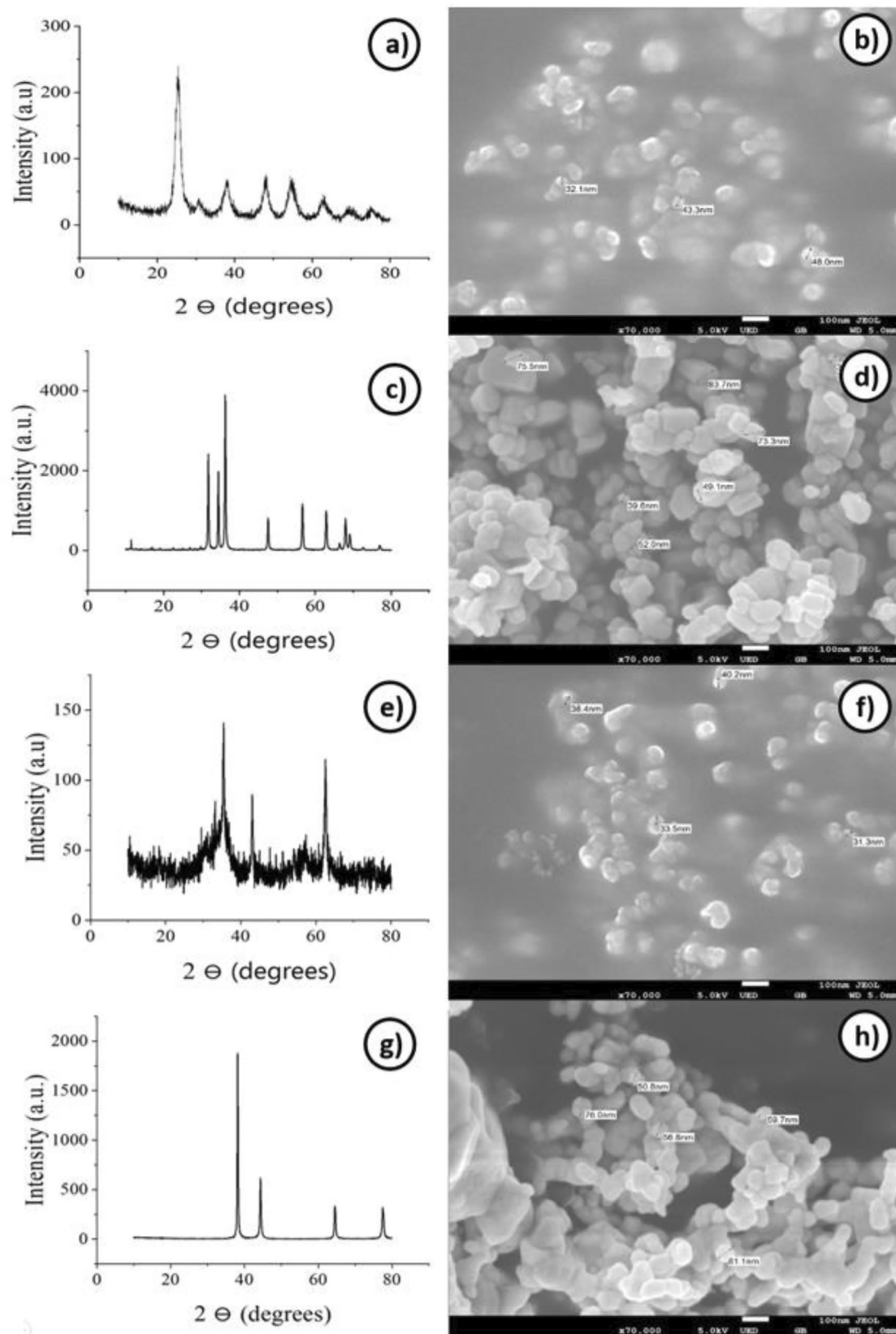


Fig. 1 Characterization of NPs: TiO₂-NPs. (a) XRD pattern, and (b) SEM/EDS image; ZnO-NPs. (c) XRD pattern, and (d) SEM/EDS image; Fe₃O₄-NPs. (e) XRD pattern, and (f) SEM/EDS image; and Ag-NPs. (g) XRD pattern, and (h) SEM/EDS image

Table 1 The physicochemical properties of soil treated with 200 g ha⁻¹ of NPs and cultivated with wheat seeds in land conditions after destructive sampling (60 and 130 DAS)

	Control	TiO ₂ -NPs	ZnO-NPs	Fe _x O _x -NPs	Ag-NPs
pH_60	7.5±0.01 bc	7.4±0.04 c	7.7±0.02 a	7.7±0.02 a	7.6±0.03 ab
pH_130	8.7±0.13 a	8.8±0.06 a	8.7±0.01 a	8.8±0.04 a	8.8±0.04 a
EC_60 (d Sm ⁻¹)	0.6±0.02 ab	0.7±0.03 a	0.5±0.03 c	0.5±0.01 bc	0.7±0.04 a
EC_130 (d Sm ⁻¹)	0.3±0.09 a	0.1±0.00 a	0.1±0.00 a	0.1±0.01 a	0.1±0.03 a
SOM_60 (%)	4.7±0.33 a	4.4±0.11 a	5.2±0.06 a	4.9±0.12 a	4.6±0.08 a
SOM_130 (%)	5.9±0.01 a	4.7±0.25 c	6.3±0.07 a	5.9±0.06 ab	5.1±0.27 bc
K_60(gkg ⁻¹)	2.4±0.01 a	2.2±0.00 ab	1.9±0.01 b	1.9±0.02 bc	1.5±0.00 c
K_130 (g kg ⁻¹)	2.9±0.00 b	2.8±0.02 b	2.4±0.00 b	3.6±0.02 a	3.4±0.00 a
P_60 (gkg ⁻¹)	0.3±0.00 b	0.4±0.00 a	0.2±0.00 c	0.2±0.00 c	0.4±0.00 a
P_130 (g kg ⁻¹)	0.4±0.00 c	0.5±0.00 b	0.3±0.00 d	0.4±0.00 c	0.6±0.00 a
N_130 (g kg ⁻¹)	2.9±0.00 b	2.4±0.13 d	3.2±0.03 a	2.9±0.03 b	2.6±0.13 c
C_130 (g kg ⁻¹)	86.9±0.08 b	68.9±3.68 e	91.6±0.95 a	85.5±0.92 c	74.9±3.92 d
TOC_130 (g kg ⁻¹)	34.8±0.03 b	27.6±1.47 d	36.6±0.38 a	34.2±0.37 b	29.9±1.57 c
Dw (Mg ha ⁻¹)	13.9±0.34 c	14.7±0.17 bc	18.3±0.50 a	14.7±0.08 bc	15.4±0.08 b
Yield (Mg ha ⁻¹)	6.7±0.49 bc	4.3±0.26 d	11.5±0.48 a	7.1±0.17 b	5.3±0.08 cd

Different lower-case letters in each column indicate statistical differences between treatments ($p \leq 0.05$); EC electrical conductivity; SOM soil organic matter; K, P, N, and C correspond to total potassium, phosphorus, nitrogen or carbon, respectively; TOC total organic carbon; DW dry biomass (plant) weight

The phosphorus (P) concentration at 60 DAS was higher in the TiO₂-NPs treatment (0.4 ± 0.00 g kg⁻¹) and lower in the ZnO-NPs treatment (0.2 ± 0.00 g kg⁻¹) compared to the control treatment (0.3 ± 0.00 g kg⁻¹). At 130 DAS the highest concentration was observed in the Ag-NPs treatment (0.6 ± 0.00 g kg⁻¹), while the lowest was in the ZnO treatment (0.3 ± 0.00 g kg⁻¹), both compared to the control treatment (0.4 ± 0.00 g kg⁻¹).

At the end of the growth cycle (130 DAS), the dry biomass of the plant treated with ZnO-NPs (18.3 ± 0.50 Mg ha⁻¹) was highest compared with the control treatment (13.9 ± 0.34 Mg ha⁻¹). The wheat grain yield was higher in the ZnO-NPs treatment (11.5 ± 0.48 Mg ha⁻¹), while the lowest yield was observed in the TiO₂ treatment (4.3 ± 0.26 Mg ha⁻¹; Table 1).

The length of the spike (Fig. 2a) showed the best length in Fe_xO_x -NPs treatment (4.54 ± 0.17 cm) compared with

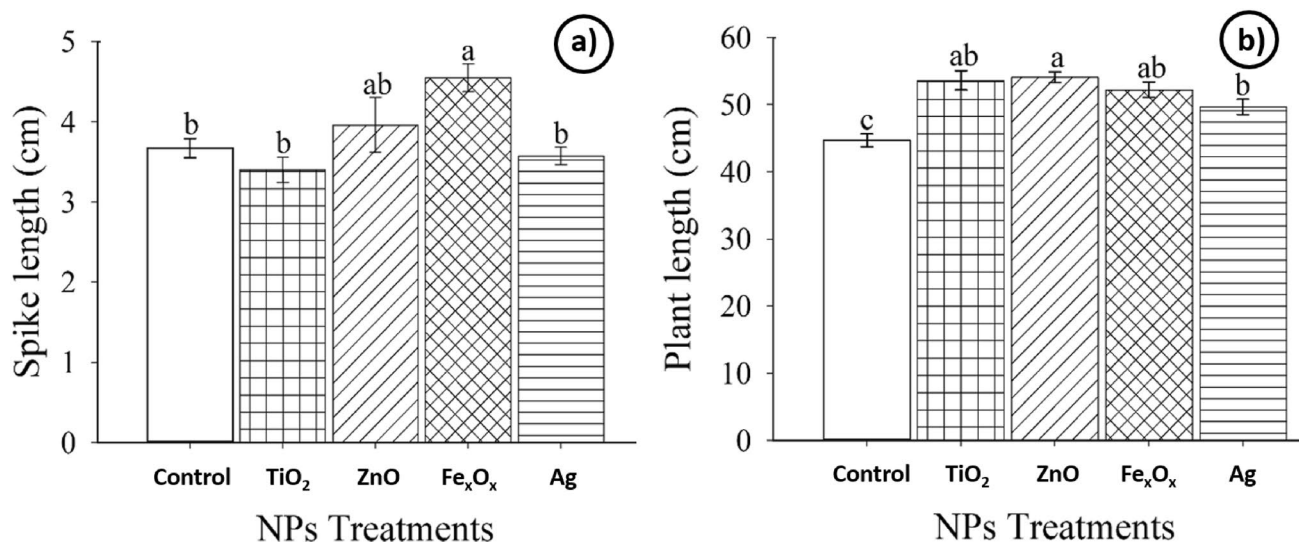


Fig. 2 Plant characteristics of wheat (*Triticum durum* Desf.) cultivated in land conditions after the treatment with 200 g ha⁻¹ of different NPs (untreated–control treatment-, TiO₂, ZnO, Fe_xO_x, or Ag).

(a) Wheat spike length (cm) and (b) Wheat plant length (cm). Different lower-case letters in figure indicate statistical differences between treatments

control treatment (3.67 ± 0.12 cm). The length of the plant (Fig. 2b) was better in ZnO -NPs treatment (54.06 ± 0.78 cm) compared to control treatment (44.68 ± 0.96 cm).

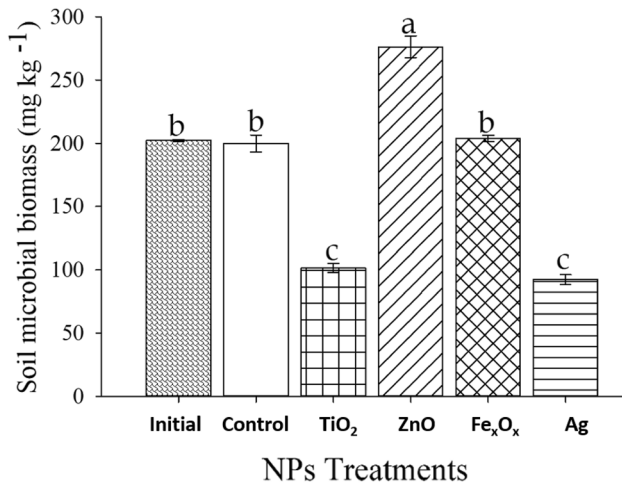


Fig. 3 Soil microbial biomass from soil cultivated with wheat (*Triticum durum* Desf.) in land conditions and treated with 200 g NPs ha⁻¹ of TiO₂, ZnO, Fe_xO_x, or Ag. Besides, an unamended treatment was used as (control), while the condition at the experiment onset was called ‘initial’ treatment. Different lower-case letters in each bar indicate statistical difference between treatments

The soil treated with ZnO -NPs exhibited the highest levels of soil microbial biomass (276.27 ± 8.63 mg C kg⁻¹ soil), as witnessed in Fig. 3, compared with Control treatment (199.91 ± 6.70 mg C kg⁻¹ soil) and initial condition (202.25 ± 0.67 mg C kg⁻¹ soil).

ICP-OES analysis (Table 2) showed the basic elements of interest in this study (Ti, Zn, Fe, and Ag), Ag element concentration is present in soil only in the treatment with these NPs (2.7 ± 0.18 mg kg⁻¹) compared with initial conditions (2.0 ± 0.00 mg kg⁻¹) and was not detectable in tissues. Ti concentration (57.2 ± 0.6 mg kg⁻¹) was higher in soil with TiO₂ -NPs and ZnO-NPs (58.3 ± 0.7 mg kg⁻¹) compared with initial (51.3 ± 0.6 mg kg⁻¹). Besides, Ti was translocated to the root. Zn concentration increased in soil with ZnO-NPs (67.3 ± 0.6 mg kg⁻¹), compared to initial values (55.7 ± 0.6 mg kg⁻¹) and showed translocation to the grain. Fe concentration increased in soil with ZnO ($12,559.0 \pm 29.7$ mg kg⁻¹) and Fe_xO_x ($12,449.7 \pm 73.9$ mg kg⁻¹)-NPs, compared to initial concentration ($10,346.7 \pm 0.6$ mg kg⁻¹) and control treatment ($11,948.3 \pm 37.3$ mg kg⁻¹). Besides, Fe was translocated to the tissues as witnessed in Table 2.

Table 2 Content of elements in soil, vegetable tissues, and grain of wheat (*Triticum durum* Desf.) cultivated in land conditions after the treatment with 200 g ha⁻¹ of different NPs (untreated–control treatment-, TiO₂, ZnO, Fe_xO_x, or Ag), according to the ICP-OES analysis

Treatment		Fe (mg kg ⁻¹)	Ag (mg kg ⁻¹)	Ti (mg kg ⁻¹)	Zn (mg kg ⁻¹)
Initial (At the onset of the experiment)	Soil	10,346.7 ± 0.6 e	2.0 ± 0.00 c	51.3 ± 0.6 c	55.7 ± 0.6 c
	Control				
Control	Soil	11,948.3 ± 37.3 d	2.0 ± 0.01 c	55.2 ± 0.6 a	57.1 ± 0.6 c
	Root	3582.2 ± 0.4 d	ND	52.1 ± 0.5 c	54.0 ± 0.6 a
	Shoot	245.6 ± 0.3 c	ND	37.2 ± 0.6 a	57.1 ± 0.6 a
	Grain	47.2 ± 0.2 d	ND	36.3 ± 0.6 a	55.1 ± 0.5 d
TiO ₂ NPs	Soil	12,344.3 ± 63.3 bc	2.5 ± 0.09 ab	57.2 ± 0.6 a	62.3 ± 0.7 b
	Root	5446.0 ± 0.1 a	ND	55.3 ± 0.4 b	51.0 ± 0.6 b
	Shoot	146.1 ± 0.6 e	ND	11.2 ± 0.6 c	48.2 ± 0.6 b
	Grain	146.3 ± 0.7 a	ND	07.2 ± 0.6 b	74.1 ± 0.6 a
ZnO NPs	Soil	12,559.0 ± 29.7 a	2.2 ± 0.04 bc	58.3 ± 0.7 a	67.3 ± 0.6 a
	Root	4601.0 ± 0.6 c	ND	60.0 ± 0.6 a	50.1 ± 0.6 b
	Shoot	337.1 ± 0.6 a	ND	38.0 ± 0.6 a	40.3 ± 0.7 c
	Grain	43.2 ± 0.6 e	ND	35.1 ± 0.6 a	75.1 ± 0.6 a
Fe _x O _x NPs	Soil	12,449.7 ± 73.9 ab	2.0 ± 0.06 c	50.9 ± 0.9 b	61.3 ± 0.6 b
	Root	3013.9 ± 0.2 e	ND	27.3 ± 0.7 d	32.1 ± 0.6 c
	Shoot	238.0 ± 0.2 d	ND	13.0 ± 0.6 bc	26.1 ± 0.6 e
	Grain	56.1 ± 0.7 c	ND	08.1 ± 0.6 b	60.1 ± 0.6 c
Ag NPs	Soil	12,151.0 ± 13.6 cd	2.7 ± 0.18 a	49.3 ± 0.6 b	61.3 ± 0.7 b
	Root	4758.7 ± 0.9 b	ND	23.2 ± 0.6 e	49.1 ± 0.6 b
	Shoot	313.3 ± 0.4 b	ND	14.2 ± 0.6 b	30.1 ± 0.6 d
	Grain	66.7 ± 0.9 b	ND	06.9 ± 0.5 b	65.2 ± 0.6 b

Different lower–case letters in each column indicate statistical differences between treatments ($p \leq 0.05$). ND not detectable

Discussion

The soil properties such as bulk density, texture, N, C, and TOC had no statistical differences between the treatments compared to initial and control measures at 60 and 130 DAS. Table 1 shows the physicochemical properties of soil after each destructive sampling (60 and 130 DAS), the dry biomass of wheat plants, and grain yield at 130 DAS.

At 60 DAS, the pH was lower in the TiO₂ treatment, with the highest pH recorded in the ZnO and Fe_xO_x treatments. No significant differences were observed between the treatments by the end of the harvest period (130 DAS). Nevertheless, the increase in pH from the initial to the final condition was attributed to crop irrigation, according to Masseroni et al. (2021).

The EC at 60 DAS was lower in the ZnO treatment when compared to the control treatment, probably because the NPs were combined with other components or minerals in the soil (García-Gómez et al. 2015). Besides, electrolytes of ZnO-NPs can increase de EC by its ionic conductivity which is around 0.009 S cm⁻¹. In addition, the EC may be higher than at the initial values because of the dissolution and solubilization process of NPs (García-Gómez et al. 2014; Zahra et al. 2019). By the end of the growth period (130 DAS), no significant EC differences were observed among the treatments. Nevertheless, it exhibited a general decrease compared to the initial average value.

At 60 DAS, soil organic matter exhibited no significant differences among treatments; nonetheless, it was notably higher than the initial average percentage. As the harvest concluded (130 DAS), it was observed that the treatments involving TiO₂ and Ag -NPs had lower levels of organic matter than the control treatment. This decrease could be attributed to inhibited root growth and limited adsorption of organic matter in the soil due to the adverse effects of these NPs on soil microorganism metabolism. Furthermore, in calcareous soils, these NPs reduce the activities of urease and phosphatase enzymes. Additionally, the antimicrobial properties of these NPs may have contributed to a decline in microbial activity within the soil (Huang et al. 2022). In summary, the organic matter content increased overall by the end of the cycle compared to the initial values.

The potassium (K) concentration at 60 DAS was lower in the Ag-NPs treatment compared to the control treatment. By 130 DAS, the highest total potassium concentration was observed in the Fe_xO_x and Ag NPs treatment, consistent with the studies reported by Jhazab et al. (2015) and Guzman-Baez, et al. (2021). These researchers studied similar conditions when they growth wheat using different doses of a silver NPs. They found a better K use efficiency in the grain and result on better yield compared with the control treatment in low doses (25–50 ppm).

The phosphorus (P) concentration at 60 DAS was higher in the TiO₂-NPs treatment and lower in the ZnO-NPs treatment compared to the control treatment. Various studies suggest a chemical interaction between phosphate and active sites of NPs, also the cations of NPs can make complexes and precipitates with phosphates; Moharami and Jalali (2015) and Sun et al. (2021) concluded that the presence of NPs decreases the bioavailability of phosphates in soils. Besides, ZnO-NPs interact directly with the phosphate and form zinc phosphate insoluble aggregates that prevent the leaching of phosphates (Rathnayake et al. 2014; Verma et al. 2022; Yusefi-Tanha et al. 2020).

Upon completion of the growth cycle, it was determined that the dry biomass of the plant treated with ZnO-NPs exceeded that of the control treatment. This is attributed to Zn's dual role as a micronutrient and antioxidant, which facilitates the plants' absorption of ammonium nitrogen. In addition, these ZnO-NPs enhance chlorophyll activity and the assimilation of nutrients such as Zn, B, Cu, K, and Fe. These materials also promote microbial activity in the rhizosphere and stimulate the production of enzymes, including phosphatase and phytase, while mitigating the plant's hydric stress (Tian et al. 2019).

The wheat grain yield was higher in the ZnO-NPs treatment, while the lowest yield was observed in the TiO₂ treatment. The yield obtained was higher than that INIFAP reported (8.5 Mg ha⁻¹) in similar crop conditions. The lower yield reported in this study (TiO₂-NPs treatment) was attributed to the antimicrobial activity and the low growth of the spike (Cox et al. 2016; El-Temsah and Joner 2012).

The length of the spike (Fig. 2a) determines the amount of wheat seeds the spike can hold. According to this study, Fe_xO_x-NPs yielded the best results as they effectively increased the iron levels in the soil, improved the plant's root system, enhanced the iron content in the plant, and boosted chlorophyll levels. Furthermore, these NPs have the potential to enhance the metabolism of microorganisms, promote growth, and stimulate germination (Saleem and Zaidi 2020).

As depicted in Fig. 2b, the plant length (cm) exhibited more significant growth in the soil treated with ZnO-NPs than in the control. This growth could be attributed to the properties of ZnO, which have the potential to convert it into a valuable micronutrient (Zn). The calcareous soil prevalent in this region is known to have a deficiency of this essential element, which can be effectively adsorbed in the soil clay and made readily bioavailable for the growth of plants (García-Gómez et al. 2018; Surendranath and Mohanan 2021). The soil treated with ZnO-NPs exhibited the highest levels of soil microbial biomass (Fig. 3). These NPs are known to stimulate plant growth and enhance microbial activity in the rhizosphere (Masseroni et al. 2021). Conversely, the soil treated with TiO₂-NPs showed

the lowest abundance of soil microbial biomass as a result of their antimicrobial characteristics in the soil (Cox et al. 2016; El-Temsah and Joner 2012).

In the southeastern region of Coahuila, the soil is composed of calcareous material that is deficient in essential nutrients. The findings of this study indicate that plant growth in untreated soil did not exhibit any significant improvements in length or weight, while soil treated with NPs yielded positive results. Furthermore, the formation of carbonates on the soil's surface can hinder the absorption of vital nutrients and water, as documented by previous studies (García-Gómez et al. 2018).

Table 2 shows the movement of the elements from the soil through the plant. The initial measurement of soil was to determine the amount of each element without rinse or sown conditions and how they translocate. The Ag element increased only in the soil where the Ag NPs were sprayed; there was no Ag translocation in the tissues. TiO_2 NPs increase the nutrients like N, P, and Fe in soil, but they do not have an effect on Zn or K, stimulate the soil microflora; at this concentration (low), these NPs are stimulants of soil nutrients and microbial dynamics (Kaur et al. 2022). Adding ZnO NPs to the soil could increase the microbial diversity and richness of some metal-tolerant bacteria, which could improve the uptake of different nutrients because of the metabolism of these bacteria. ZnO-NPs help mitigate oxidative stress and increment the Zn bioavailability in soil (Huang et al. 2022). Fe is an essential metal for plants; adding Fe_xO_x NPs contributes to assimilating iron like a micronutrient and accumulates more in the soil where the roots are; in the same way, studies demonstrated that NPs of iron are commonly uptake by plants as the same with Zn NPs in shoots and leaves and Fe NPs is the most uptake by roots (Jia et al. 2023).

This study can conclude that the observed increase in SOM, pH, and EC levels may be attributed to the irrigation period during the growing wheat period and the addition of ZnO-NPs. These nanoparticles have been found to facilitate growth and stimulate soil microbial activity. Upon completion of the harvest, the total yield in soil treated with ZnO NPs surpassed the typical yield reported by INIFAP. This can be ascribed to the NPs' growth-promoting qualities, ability to transform into micronutrients for the plant, and function as antioxidants. The rise in soil microbial biomass growth is likely attributable to the activity and characteristics of the ZnO-NPs. Conversely, the lower microbial abundance of soil treated with TiO_2 -NPs or Ag-NPs may be due to both TiO_2 -and Ag-NPs having antimicrobial properties. Fe_xO_x -NPs increased the crop characteristics and yield compared to the control treatment. Still, the effect was slightly evident, maybe because wheat is not a highly demanding crop, unlike some legumes. Therefore, adding ZnO-NPs to the leptosol improves wheat yield and soil

microbial biomass. It could be necessary to carry out long-term studies to know the residual effect of some NPs on agricultural soil and rural ecosystems and elucidate the collateral effect of NPs spreading in worldwide land.

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Declarations

Conflict of interest Not applicable.

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