

The Effect of Foliar Application of ZnO Nanoparticles and ZnSO₄ on Improving the Growth and Production of Durum Wheat

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Abstract. A factorial experiment was carried out by foliar application of zinc in two forms zinc sulphate (ZnSO4) and zinc oxide nanoparticles (ZnO NPs) prepared from aqueous zinc acetate using a wet chemistry method, which were characterized using an atomic force microscope (AFM) as having dimensions of 40-80 nanometres. The foliar of two zinc forms were applied in three concentrations (25, 50 and 100 ppm) in addition to distilled water with the aim of studying their effect on the growth and productivity of durum wheat plants (Triticum durum, L var. Sham 7) grown under the conditions of Deir Ez-Zor Governorate-Syria. The results showed that adding zinc had a significant effect on all tested morphological indicators (plant heights - chlorophyll - number of branches/plant - number of spikes /plant) and productivity (1000 kernel weight, grain and straw productivity), as well as the grain content of some elements (nitrogen, phosphorus, potassium and zinc). The treatments for adding zinc oxide nanoparticles were significantly superior to those of zinc sulphate in all tested indicators. as the lowest values for the tested indicators were recorded in the regular zinc sulphate treatment at a concentration of 25 ppm compared to the rest of the treatments except for the control treatment, while the Nano zinc oxide treatments at a concentration of 50 and 100 ppm showed superiority significantly in all studied indicators especially in increasing wheat productivity by 24-26%, compared to the control treatment.

Keywords. Durum wheat, Foliar application, Zinc oxide nanoparticles (ZnO NPs), Zinc sulphate(ZnSO₄).

1. Introduction

The agricultural sector faces many challenges, including climate change, increased consumption of agricultural products, and shrinking cultivated area, which necessitates the advancement of agricultural development to achieve economic stability. Hence the importance of using nanotechnology, which enables the development of modern methods to find and address many agricultural problems. Thus, advancing the agricultural field, which achieves a great impact in serving society and developing the environment, as scientists and researchers began to think about nanotechnology as a result of the recession, the decrease in crop yields, the decrease in organic



materials, the shrinkage of arable land, and the decrease in the availability of water. Nano applications in the agricultural field the most important mechanism for achieving modern agricultural methods, which are summarized in the low economic cost resulting from the absence of epidemic diseases that affect various strategic crops (cereals, for example), and increased the efficiency of manufactured fertilizers with their low material cost, and resistance to agricultural product for different environmental conditions [1].

The wheat crop (*Triticum durum*, *L*.) belongs to the Poaceae family, which constitutes an important nutritional value represented by the good balance in its grains between proteins and carbohydrates, as well as it's containing quantities of fats, vitamins, some mineral salts, and the amino acids it needs the human in his food [2,3].

Foliar addition means spraying nutrients in the form of solutions on the vegetative system, and it can provide the plant with 85% of its nutrient needs [4]. In addition to ensuring a rapid response to the absorption of nutrients from the vegetative parts of the plant [5], this method is economical by reducing the need for large quantities of nutrients [6].

Zinc is considered an essential micronutrient for humans, animals and plants, foliar application of ZnO NPs has shown to have a positive effect on growth and plant physiology, and foliar fertilization with relatively low amounts of modified Zn Nano fertilizers has shown an increase in yield, fruit quality, juice sugars and maturity index [7]. It was found [8] that the use of chelated zinc (Zn DTPA) led to an increase in wheat grain yield by 44.4% compared to the control without addition.

One of the important applications of nanotechnology in the agricultural field is the use of Nano fertilizers, which contribute to the slow and gradual release of fertilizers due to the ability of Nano grains to retain the fertilizer material for a longer period due to the higher surface tension of these granules compared to traditional surfaces. This slow release has contributed to increasing the effectiveness of nutrient absorption by the plant [9].

Afshar *et al.* [10] indicated that adding Nano zinc oxide at five levels (0, 24, 36, 48, and 60 g. ha⁻¹) sprayed on wheat led to an increase in growth indicators and yield, and was superior to the level treatment (60 g. ha⁻¹) over The rest of the levels are in the number of ears m⁻², the number of grains per ear, and the weight of 1000 grains. [11]. showed that spraying zinc Nano fertilizers (ZnO NPs) at a level of 25 ppm and for two stages of wheat growth led to the superiority of Nano zinc in indicators. All growth, including: length of the stem, roots, number of leaves in the plant, dry weight, wet weight of the root and vegetative systems, number of branches, number of spike m⁻², number of grains per spike, 1000 kernel weight, and grain yield compared to not adding.

This research was carried out in order to determine the optimal concentration of foliar application of Zinc forms (ZnO NPs and ZnSO₄) and their role in the growth and productivity of durum wheat (*Triticum durum*, L var. Sham 7) growing under the conditions of Deir Ezzor Governorate-Syria.

2. Materials and Methods

The research was carried out at the Agricultural Scientific Research Centre - Saalo Research Station, 30 km east of Deir Ezzor (longitude 35.22 east, latitude 40.11 north, and an altitude of 203 meters above sea level) in the 2021/2022 agriculture season. The land was cultivated and divided into experimental plots of 2 m^2 , and a randomly compound soil sample was taken within the experimental plots before planting at a depth of 0-30 cm, and transferred to the soil laboratory in the Soil and Land Reclamation Department, Faculty of Agriculture at Al-Furat University, to determine some physical and Chemical properties (Table 1).

Table 1. Physical and Chemical properties of experiment soil before planting.

	Faa	OM	CaCO		Ava	ilable Nutrients	
pН	de m ⁻¹	0/v1 0/		Ν	Р	K	Zn
	us.m	/0	/0	%	ppm	ppm	ppm
7.8	2.32	1.07	22.5	0.16	7.5	406	0.95
Particle	e size distril	oution%	Call tantan		Bulk density	Particle density	Total porosity
Clay	Silt	Sand	Son texture		g/cm ³	g/cm ³	%
46	22	32	Clay		1.45	2.45	40.81

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Pure zinc oxide nanoparticles powder (ZnO NPs) were prepared in the Nano laboratory of the Department of Physics, Faculty of Science - Damascus University, by wet chemistry method using aqueous zinc acetate [Zn (CH₃ COO)₂.2H₂O], atomic force microscope (AFM) and scanning electron microscopy (SEM) was described, and the particle size distribution was measured by dynamic light scattering (DLS), and the elemental ratio was determined by elemental analysis by Spectroscopy Energy Dispersive X-Ray Analysis (EDX).

Seeds of durum wheat, *Triticum durum* L. (Sham 7 variety), were sown at a rate of 200 kg ha⁻¹. Mineral fertilizers (NPK) were added according to the fertilizer recommendation of the Ministry of Agriculture and Land Reclamation for irrigated wheat cultivation. The zinc sulphate and zinc oxide nanoparticles were added as foliar addition (whole plant wetting) at three levels (25, 50 and 100 ppm) addition to distilled water in three successive batches after 30, 60 and 90 days from the completion of germination. After reaching full growth, ten plants were randomly selected from each experimental plot for morphological measurements. Plant height was measured at harvesting stage using a ruler extended from the base to the top of the plant. The number of branches per plant was counted, and the average count was calculated. Total chlorophyll content was measured using a field chlorophyll meter (SPAD) at the beginning of the heading stage. The number of spikes per plant was counted, and the average weight of 1000 grains was estimated from each experimental plot. The average weight of 1000 grains was estimated from each experimental plot and for each concentration with four replicates. The grains content of nitrogen, phosphorus, and potassium were determined according to [12-14].

The field experiment was carried out according to the factorial experiment, randomized complete block design, with two factors (Zinc form and level of addition), with four replications for each treatment. The data were analysed after statistical collection and tabulation using the M-Stat-C statistical analysis program to calculate the Least Significant Difference (LSD) values, at the 0.05 level.

3. Results and Discussion

3.1. Characterization of ZnO NPs

The surface topology and particle size distributions of the ZnO was evaluated using Atomic Force Microscopy (AFM, Nanosurf easyScan2, Switzerland) as showed in Figure 1. We notice from the AFM image that the formed nanoparticles take a homogeneous spherical shape, dimension distribution, and granule density, as shown by the distribution curves for the diameters of the granules measured by the AFM, and the sizes of the granules vary within the range of 40 nm to 80 nm (Figure. 1)







Figure 2 illustrate the SEM image of the prepared ZnO particles. The ZnO-NPs prepared was in spherical and agglomerated with diameter about 60 nm. The EDX spectra presented in Figure (3), including the atomic percentage concentrations is presented in the table included in the insets of Figure 2. EDX analysis results confirm that the synthesized ZnO Nano powder are made of Zn and oxygen with an atomic stereometric ratio of approximately equal to one. No other impurity peak was observed in the spectrum, up to the detection limit of EDS.



Figure 2. SEM image and Elemental composition of of Zinc Oxide Nano powder.

The particles size distribution graph obtained from the DLS of zinc oxide Nano powder presented in figure 3. The number-based distribution revealed narrow scattering centers and monodispersity with a mean diameter of 35 nm. These above results showed that as prepared ZnO powder were in the Nano range.



Figure 3. Number size distribution of Zinc Oxide nanoparticles.

3.2. Morphological Characteristics of Wheat Plants

The results in Table No. (2) indicate that there is a significant superiority in the studied morphological characteristics (number of spikes and number of branches in the wheat plant and values of chlorophyll) by using foliar application of zinc oxide nanoparticles compared with treatments of regular zinc sulphate at all applied concentrations. However, there was no significant difference in plant length at harvesting stage.

Table 2. Effect of Zinc material type on some morphological indicators of wheat plants.

Materials	Plant length (cm) harvesting stage	No. of spikes/ plant	No. of branches/ plant	Chlorophyll SPAD
ZnSO ₄	90.0 ^a	38.12 ^b	3.77 ^b	4.77 ^b
ZnO NPs	90.69^{a}	38.94 ^a	4.49^{a}	5.46^{a}
LSD 0.05	0.717	0.482	0.382	0.382

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On the other hand, regardless of the type of material used, it is noted that there is a significant superiority of the different applied levels of Zinc (25, 50 and 100 ppm) compared to the control treatment (table 3) and the highest values were recorded when foliar application at concentrations of 50 and 100 ppm for all morphological tested traits.

Table 3. Effect of different Zinc levels application on some morphological indicators of wheat plants.

Zinc levels ppm	plant length (cm) Harvest stage	Chlorophyll SPAD	No. of branches/ plant	No. of spikes/ plant
0	87.00 ^b	33.33°	2.65 ^b	3.65 ^b
25	91.19 ^a	39.84 ^b	4.58 ^a	5.51 ^a
50	91.75 ^a	40.91 ^a	4.53 ^a	5.53 ^a
100	91.44 ^a	40.83 ^a	$4.78^{\rm a}$	5.78^{a}
LSD 0.05	1.014	0.682	0.54	0.54

Table (4) shows the combined effect of the type of substance (zinc sulphate and zinc nanoparticles) and the with different levels (25, 50 and 100 ppm) on tested morphological traits of wheat plants. It is noted that there is a significant superiority of the addition of zinc sulphate and zinc oxide compared with control treatment. On the other hand, the two treatments with a concentration of 50 and 100ppm of zinc oxide nanoparticles were the most effective in the studied traits.

Table 4. Effect of different levels of zinc sulphate and zinc nanoparticles on some morphological indicators of wheat plants.

	ZnO	plant length (cm)	Chlorophyll		No. of spikes/plant
Materials	conc. ppm	Harvesting	SPAD	No. of branches/ plant	
	0	87.00 ^d	33.33 ^d	2.65 ^d	3.65 ^d
7.90	25	90.53°	39.44 ^{bc}	4.10°	5.10 ^c
$ZnSO_4$	50	91.38 ^{abc}	40.58^{a}	4.06°	5.05 ^c
	100	91.38 ^{abc}	39.13 ^c	4.28 ^{bc}	5.28 ^{bc}
	0	87.00^{d}	33.33 ^d	2.65^{d}	3.65 ^d
	25	91.00 ^{bc}	40.25^{ab}	5.05 ^{ab}	5.93 ^{ab}
ZIIO NPS	50	92.13 ^{ab}	41.24 ^a	5.00 ^{ab}	6.00^{ab}
	100	92.78^{a}	$40.94^{\rm a}$	5.28^{a}	6.28 ^a
LSD 0.	05	1.43	0.964	0.764	0.764

Figure No. (4) shows the percentages of the increase in the studied traits as a result of the addition of zinc sulphate and zinc nanoparticles at different concentrations compared with the control treatment (without any addition of zinc).



Figure 4. The Percentage increase on the tested morphological indicators of durum wheat at different levels application of zinc sulphate and zinc oxide nanoparticles (compared with control treatment).



The previous figure shows a clear increase in the average length of the plant at the harvesting stage compared to the control, which ranged between 4 and 7%, and an increase in the average content of chlorophyll ranged between 18 and 23%, as well as an increase in the average number of branches ranging between 43 and 99%, and the increasing in number of spikes was 40 and 72%. The figure also shows that the addition of zinc oxide nanoparticles by foliar gave better results than zinc sulphate. The values of studied morphological indicators increased with increasing the level application of ZnO NPs up to 50 ppm, where the differences were not significant between the concentration 50 and 100 ppm. This may be due to the fact that spraying with foliar nutrients contributes to a significant increase in most of the characteristics of vegetative growth, and that increasing the absorption of nutrients spraved through the leaves leads to regulating the nutritional balance, which is reflected in plant growth, and regulating the hormonal content, as the photosynthesis process is activated. Energy production and protein synthesis within plant tissues, which leads to plant growth [15-17], these results agree with those findings of [18]. Thus, the observed increases in plant growth are due to the role of ZnO NPs in increasing chlorophyll content because it is a common indicator of photosynthesis efficiency on the plant, which is one of the most important indicators of growth [19], and this was reflected in the increase in the number of branches/plant and the number of spikes/plant.

3.3. Productivity Characteristics of Wheat Plants

The results in Table No. (5) indicate that there is a significant superiority in the studied productivity characteristics (grain productivity, straw and 1000 kernel weight) by using foliar application of zinc oxide nanoparticles compared with treatments of regular zinc sulphate at all applied concentrations.

Materials	Grain productivity (ton .h ⁻¹)	Straw (ton .h ⁻¹)	1000 kernel weight (g)
ZnSO ₄	7.82 ^b	8.19 ^b	50.52 ^b
ZnO NPs	8.28^{a}	8.72^{a}	51.95 ^a
LSD 0.05	0.023	0.042	0.588

Table 5. Effect of Zinc material type on some productivity indicators of wheat plants.

Regardless of the type of material used, it is noted that there is a significant superiority of the different applied levels of Zinc (25, 50 and 100 ppm) compared to the control treatment (table 6) and the highest values were recorded when foliar application at concentrations of 100 ppm for all productivity tested traits.

Zinc levels Ppm	Grain productivity (ton .h ⁻¹)	Straw (ton .h ⁻¹)	1000 kernel weight (g)
0	7.07^{d}	7.41 ^d	49.49 ^c
25	8.17°	8.55 ^c	51.36 ^b
50	8.41 ^b	8.85^{b}	51.83 ^{ab}
100	8.55 ^a	9.01 ^a	52.25 ^a
LSD 0.05	0.111	0.06	0.83

Table 6. Effect of different Zinc levels application on some productivity indicators of wheat plants.

Table (7) shows the combined effect of the type of substance (zinc sulphate and zinc nanoparticles) and the with different levels (25, 50 and 100 ppm) on tested productivity traits of wheat plants. There was a significant superiority of zinc sulphate and zinc oxide nanoparticles with all levels of addition in all studied productivity indicators (productivity, straw and weight of 1000 kernel) compared to the control treatment (without addition), and the two treatments the concentration of 50 and 100ppm were the most effective in the studied traits.

25

50

100

ZnO NPs



51.71^{bc}

52.80^{ab}

53.78^a

Materials	Zn (ppm)	Productivity (ton .h ⁻¹)	Straw (ton .h ⁻¹)	1000 kernel weight(g)
	0	7.07 ^g	7.41 ^f	49.49 ^d
ZnSO ₄	25	7.93 ^f	8.23 ^e	51.02 ^c
	50	8.07^{e}	8.44^{d}	50.85 ^c
	100	8.22^{d}	8.68 ^c	50.72 ^c
	0	7.07 ^g	7.41 ^f	49.49^{d}

8.86^b

9.26^a

9.34^a

8.42^c

8.75^b

8.87^a

Table 7. Effect of different levels of zinc sulphate and zinc	e nanoparticles on some productivity
indicators of wheat plant	ts.

LSD 0.05 0.045 0.084 1.175 Figure (5) shows the percentages of the increase in the studied traits as a result of the addition of zinc sulphate and zinc oxide nanoparticles, at different concentrations compared to the control treatment (without any addition of zinc). The figure shows a clear increase in the average grain productivity of wheat plants compared to the control ranged between 12 and 26 %, and an increase in the average produced straw ranging between 12 and 26 %, as well as an increase in the average weight of 1000 Kernel, which ranged between 3 and 9%, The figure also shows that the addition of zinc oxide NPs by foliar application gave better results than Zinc sulphate and the tested productivity indicators values increased with increasing the concentration used up to 100 ppm (ZnO NPs). The reason is due to the role of nutrients in raising the efficiency of the photosynthesis process, as the number and weight of grain depends mainly on the rate of pollination and fertilization, thus increasing the activity of vital activities within the plant and improving growth indicators and photosynthesis and then transferring its products to the growing grains, because these grains after a period from its formation, it becomes the permanent downstream in annual plants, and that the bulk of the metabolites, whether newly formed or stored, lead to an increase in the grain weight and number during the fulling stage, which improves production indicators, and these results are agree with the findings of [20-24]. They obtained an increase in all wheat yield indicators by adding Nano fertilizers microelements, as they showed that the effective role played by the nanoparticles in increasing the vital and enzymatic reactions and due to the huge surface area, which leads to an increase in the speed of the reactions and the availability of an active carrier device that helps in the transfer of nutrients from the leaves to the grains, thus increasing the materials that reach the grains and thus increasing their weight and productivity, and these agree with [25] who found that application of ZnO NPs helps to increase the grain and shoot yield of wheat significantly.



Figure 5. The Percentage increase on the tested productivity indicators of durum wheat at different levels application of zinc sulphate and zinc oxide nanoparticles (compared with control treatment)



3.4. Grain Content of Some Nutrients

The results in table (8) shows the effect of different type zinc materials in wheat grains content of some nutrients (nitrogen, phosphorus, potassium and zinc) It is noted that there is a significant superiority of addition zinc oxide nanoparticles in the grain content of nitrogen, phosphorus and zinc compared to Zinc sulphate. On the other hand, the difference was not significant in the potassium content of grains.

Table 8. Effect of Zinc material type on wheat grains content of some nutrients.

Motoriala	Grains content				
Materials	N%	P%	K%	Zn(ppm)	
ZnSO4	2.59 ^b	0.48^{b}	0.47^{a}	12.20 ^b	
ZnO NPs	2.63 ^a	0.63 ^a	0.47^{a}	17.79 ^a	
LSD 0.05	0.005	0.005	0.015	0.17	

Aside from the effect of the source of the zinc element, whether it was regular zinc sulphate or zinc oxide nanoparticles, the concentration used had a clear effect in increasing the wheat grains' content of the tested nutrients (table 9), It is noted that there is a significant superiority of the different levels application (25, 50 and 100 ppm) and the differences in potassium grains content were non-significant on all levels of applications, compared with the control treatment.

Table 9. Effect of different Zinc levels application on wheat grain content of some nutrients.

Zinc levels	Grains content				
ppm	N%	P%	K%	Zn(ppm)	
0	2.52^{d}	0.41 ^d	0.46^{a}	4.30 ^d	
25	2.60°	0.57°	0.47^{a}	12.73 ^c	
50	2.63 ^b	0.61^{b}	0.47^{a}	18.69 ^b	
100	2.65 ^a	0.63 ^a	0.47^{a}	24.25 ^a	
LSD 0.05	0.007	0.007	0.214	0.24	

Considering the combined effect of the zinc source and concentration, as shown in table (10), it is noted that there was a significant superiority of addition zinc sulphate and zinc oxide nanoparticles in the grains content of nitrogen, phosphorus and zinc with all levels of addition, and the highest values were in 100 ppm addition level compared with other levels of addition. On the other hand, the differences in potassium grains content were non-significant on all levels of addition, compared with the control treatment.

Table 10. Effect of different levels of zinc sulphate and zinc nanoparticles on wheat grains content of some nutrients.

Motoriala	Zn		ţ		
wrateriais	ppm	N%	Р%	K %	Zn(ppm)
	0	$2.52^{\rm e}$	0.41 ^g	0.46^{a}	4.30 ^g
7-504	25	2.57^{d}	0.48^{f}	0.47^{a}	9.94^{f}
ZnSO4	50	2.58^{d}	0.51 ^e	0.47^{a}	14.54 ^e
	100	2.60°	0.53 ^d	0.47^{a}	20.02 ^c
	0	2.52^{e}	0.41 ^g	0.46^{a}	4.30 ^g
	25	2.64 ^b	0.67°	0.47^{a}	15.53 ^d
ZIIO NPS	50	2.69^{a}	0.72^{b}	0.47^{a}	22.85 ^b
	100	2.70^{a}	0.73 ^a	0.47^{a}	28.48^{a}
LSD 0.0	05	0.010	0.010	0.011	0.340

The percentage increase resulting from the use of foliar applications of zinc from two different sources and several concentrations on grains content of tested nutrients (zinc, nitrogen and phosphorus) appears in Figure (6).





Figure 6. The Percentage increase on wheat grain content of some nutrients at different levels application of zinc sulphate and zinc oxide nanoparticles (compared with control treatment).

The diagram shows that the addition of zinc oxide NPs by foliar application gave better results than Zinc sulphate, as the grain content values of tested mineral elements (N, P and Zn) were increased with increasing the concentration used up to 100ppm, but the increase was simple in the nitrogen and it ranged between 2 and 7%, and a greater increase were in its content of phosphorus ranged between 19 and 79%, while it was more evident in the grains content of zinc and ranged between 131 and 462%. These results agree with those obtained by [26&27] they mentioned that the zinc content of the grain increased when added by the foliar method and the use efficiency of microelements did not exceed 3-5% when adding to the soil, despite of use new chelated or organic sources, and the values were much higher when adding foliar on the top or mixed addition. and these agree with [22&23]. This may be due to the zinc oxide nanoparticles has a large surface area compared to regular zinc sulphate, which increases the absorption of larger amounts of zinc due to the increase in the plant's biological and structural interactions, and thus its percentage in the soil is small and in the plant is large compared to regular zinc sulphate [28].

In general, the increase in grains content of some nutrients is consistent with what was mentioned by many studies [29&31]. Where they found that Nano fertilizers positively affected in vegetative growth indicators and increased the ability to absorb elements from the soil solution, and the smallness of nanoparticles causes rapid entry and spread of them and then sticking to plant tissues and this leads to an increase in plant hormones, as well as Its role in improving nutrient utilization efficiency and increasing the amount of nutrients absorbed. and in general, the data indicate that the role of ZnO NPs is not only in improving plant growth and physiological indicators, but also in enhancing the absorption of nutrients by stimulating plant roots to actively exchange cations, which helps absorb more nutrients. [32].

Conclusions and Recommendations

Pure ZnO Nano powder without any contaminants was successfully prepared via wet chemistry method using zinc acetate dehydrate as precursor. Their surface morphology, size distribution and elemental analysis is studied using AFM, SEM, EDX and DLS. The AFM and SEM investigations have revealed that the structure and morphology of the ZnO powder is spherical, homogeneous and monodispersed with an average diameter less than 60nm, while the EDX spectrum showed that the elemental composition of these grains is zinc oxide. The average particle size was estimated 35 nm from particle size analyzer.

From our present work, it is concluded that the addition of zinc oxide Nano powder (ZnO-NPs) and Zinc sulphate (ZnSO₄) led to a clear increase in all morphological indicators, productivity and the content of some nutrients in wheat grains at all levels of application. As that zinc oxide nanoparticles exceeded the superiority clearly on Zinc sulphate in all indicators studied, On the other hand, the two



treatments of foliar application with zinc oxide nanoparticles at a concentration of 50ppm and 100ppm increased wheat yield by (24 and 26%), compared with the control treatment.

Finally, the study recommends to addition the ZnO Nano powder by foliar application at a concentration of 50ppm to fertilize the wheat crop, and carrying out several fertilization experiments with ZnO NPs for other varieties and crops.

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