

## EFFECT OF NANO FERTILIZATION, CHEMICAL AND HUMIC ACID ON THE VEGETATIVE GROWTH, CHEMICAL COMPOSITION AND OIL YIELD OF CUMINUM CYMINUM L.

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Received: Sep. 21, 2016

Accepted: Oct. 10, 2016

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**ABSTRACT:** *This experiment was carried out at a private farm near El-Mahalla: Gharbieah Governorate during the two successive seasons of 2013/2014 & 2014/2015 to study the effect of different levels of fertilization and nano-particles fertilizer on the vegetative growth, chemical composition and essential oil yield of Cuminum cyminum L. plants. Silicon and graphite nanoparticles were applied to cumin plants by spraying at three levels of 20, 40 and 60 mg/l two times. The results showed that, using 75 % NPK dose + humic acid and 60 mg/l nano graphite gave the highest values of growth parameters, (plant height, number of both branches and umbels/ plant) while the treatment of 75 % NPK dose +2g/plant humic acid and 40 mg/l nano graphite gave the highest values of chlorophyll a and b. The treated plants with 100 % NPK and 20 mg/l nano silica gave the highest values of N and P percentages. While, the highest K % and oil % were obtained by using 100 % NPK dose and 60 mg/l nano graphite. For the major identified components in the oil were p-menta-1-en 7-al from using 75% NPK +2g/plant humic acid with 40 mg/l nano silica. It can be recommended to apply 75 % NPK dose + 2g/plant humic acid and/ or 100 % NPK with 60 mg/l nano graphite for both to obtain the highest vegetative growth parameters and essential oil yield of Cuminum cyminum L.*

**Key words:** Nano fertilization, NPK fertilization, Cuminum cyminum

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### INTRODUCTION

Cuminum cyminum Linn. is an annual plant of the family-Apiaceae commonly known as cumin. The medicinal component of the plant is cumin oil extracted from the ripe fruit. In folk medicine, cumin is used as a carminative for stomach disorders, diarrhea, and colic, as well as particularly in veterinary medicine (Gruenwald *et al.*, 2004). The oil is especially used as a carminative and as a stringent (Baytop, 1989). The fruits of C. cyminum L. are used as a traditional flavouring in a number of ethnic cuisines and food industries. Moreover, cumin oil shows a high antifungal activity against various pathogenic fungi, and effective high antibacterial activity. Therefore, it is also used as a fumigant or additive in the storage of food tuffs (Li and Jiang, 2004). The cumin fruits contain

volatile oil (2-5%) that impart the characteristic aroma to the fruits (Behera *et al.*, 2004).

Application of NPK at certain rates plus organic fertilizer or humic acid gave the best results for increasing growth and yield as well as oil (%) in medicinal and aromatic plants. Such results were obtained by (Said *et al.*, 2015 on Hibiscus sabdariffa L).

Humic acid is known to improve nutrient retention in the growing media and increasing the water holding capacity (Dorer and Peacock, 1997).

Nanotechnology is considered as a potential solution for increasing the value of agriculture products and environmental problems. For example, with the use of nano particles and nano powders, researches can

produce controlled or delayed release fertilizers (Kottegoda., *et al.*, 2011).

Nanoparticles can improve the physical and chemical properties of the soil which were reflected on the plant growth (Amin *et al.*, 1999). Silica nano particles increase turgor pressure and plant size by improving water use efficiency and leaf relative water content (Rawson *et al.*, 1988).

The aim of this investigation was to study the effect of different levels of fertilization and nano- fertilizer on the vegetative growth, chemical composition, essential oil yield and components of *Cuminum cyminum* plants.

**MATERIALS AND METHODS**

This experiment was carried out at a private farm near El-Mahalla: Gharbieah Governorate during the two successive seasons of 2013/2014 & 2014/2015 to study the effect of different levels of fertilization and Nano- fertilizer on the vegetative growth, chemical composition, oil yield and components of *Cuminum cyminum* L. plants. The physical and chemical properties of the experimental soil are shown in Table (1).

This experiment included 21 treatments as follows:

**A-NPK treatments:**

- 1- NPK full dose (control).
- 2- 75 % NPK dose + humic acid (2gm/plant).
- 3- 50 % NPK dose + humic acid (4gm/plant).

**B- Nano particles treatments:**

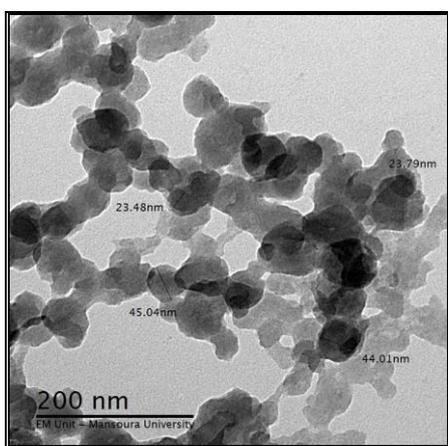
- 1. Without nano (control)
- 2- 20 mg/l silica NPs
- 3- 40 mg/l silica NPs
- 4- 60 mg/l silica NPs
- 5- 20 mg/l graphite NPs
- 6- 40 mg/l graphite NPs
- 7- 60 mg/l graphite NPs

TEM imaging of the prepared nanoparticles revealed a spherical shape of the particles, with an average size of 23.48- 45.04 nm of silicon and .20- 15.34 nm of graphite are shown in fig 1, 2.

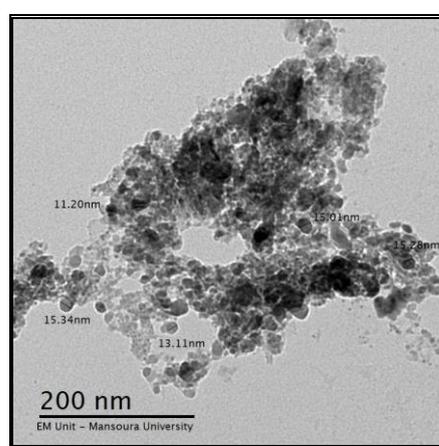
These treatments were arranged in a split plot design with three replications. NPK and humic acid treatments were randomly arranged in the main plots and nano particles concentrations were randomly distributed in the sub plots.

**Table (1): Physical and chemical analysis of the experimental soil.**

Physical Properties						Chemical Properties				
Caco3	C.sand%	F.sand%	Silt%	Clay%	Texture	Ec dsm-1	N(avail) Ppm	P(avail) ppm	K(avail) ppm	O.M%
3.57	4.12	21.52	25.11	49.25	Clayly	1.78	82.05	9.22	720	1.53



**Nano silicon  
Fig (1)**



**Nano graphite  
Fig (2)**

**Fig 1,2: TEM imaging of the prepared nanoparticles.**

Chemical fertilizers were ammonium sulphate (20.5% N), calcium superphosphate (15.5% P<sub>2</sub>O<sub>5</sub>) and potassium sulphate (48% K<sub>2</sub>O) at the rate of 150, 150 and 50 kg/fed respectively, as a full dose of NPK (recommended dose). Humic acid and calcium super phosphate were applied at one dose during the preparation of soil before planting, while ammonium sulphate and potassium sulphate were divided into two equal doses. The first dose was added after 45 days from planting, and the second one was added one month later in the two experimental seasons.

The seeds were sown in the open field on Dec 16<sup>th</sup> in the two seasons, respectively. Plants were twice sprayed with freshly prepared solution of silicon NPs and graphite NPs after 45 days from planting and then before the flowering.

The data at the end of each season were recorded on April 24<sup>th</sup> as follows:

**A- Plant growth characters:**

Plant height (cm), number of both branches and umbels/ plant, plant dry weight (g), seeds yield weight (g/plant) and weight of 1000 seeds (g).

**B- Chemical composition:**

- 1- Chlorophyll 'a', 'b' and carotenoids were described by Wellburn (1994).
- 2- The N, P and K elements were determined in the dried powdered herb according to Black (1983), Watanabe and Olsen (1965) and Richards (1954), respectively.
- 3- Volatile oil percentage was determined in ripe dry fruits according to Guenther *et al.* (1960).
- 4- The obtained volatile oil were analysed using Dschrom. Model HP-5890 with flame ionization detection that was fitted with capillary column, coated with carbowax 20 M X 0.2 min. The operating conditions were injector temperature 250° C, detector temperature 300° C. Nitrogen was used as a carrier gas with flow rate 1 ml/min, for hydrogen was 30 ml/min. The peaks were recorded and the areas under peaks were determined using HP- intergrator. Oil components were identified by comparing the

retention times with that of the authentic compounds.

**Statistical analysis:**

The obtained data were subjected to statistical analysis of variance according to Snedecor and Cochran (1980), and means separation were compared according to LSD at 5 % level.

**RESULTS AND DISCUSSION**

**1- Plant height, number of branches and number of umbels/ plant:**

Data in Tables (2 to 4) showed that plant height was significantly affected by mineral fertilizers in the first season only. Using 75% NPK dose +2g/plant humic acid gave the tallest plants, the highest number of branch and umbels/ plant with non significant differences between 100 % NPK and 75 % NPK + 2g/plant humic acid. On the other hand, using 50 % NPK dose +4g/plant humic acid recorded the least values in this respect.

It could be concluded that, using 75 %NPK +2g/plant humic acid gave the best values of plant height, both branch and umbels number which may be attributed to the role of humic acid in improving nutrients retention in the growing medium and increasing the water holding capacity (Dorer and Peacock, 1997).

Concerning nanoparticles treatments, data presented in Table (2, 3 and 4) showed that plant height, branch and umbels number were significantly affected by nanoparticles in both seasons. The tallest Table plants were resulted from the sprayed plants with all rates of either nano graphite or 20 mg/l nano silicon in the first and second seasons, respectively while the highest number of umbels/ plant were obtained by using 20 and 40 silica NPs in the 1<sup>st</sup> and 2<sup>nd</sup> seasons, respectively. On the other hand, the shortest plants, the lowest branch and umbels number were obtained by 60 mg/l nano silicon in the 1<sup>st</sup> season and the control treatment in the 2<sup>nd</sup> season.

Table (2): Effect of different fertilization levels, nanoparticles and their interaction on plant height (cm) of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Fertilization levels	Nano particles		Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)
	Control	0	20	40	60	20	40	60	
	2013/2014 season								
100 % NPK (Control)	27.26	29.60	29.28	27.77	31.20	29.86	30.00	29.28	
75 % NPK +2g/plant humic acid	29.96	29.11	29.23	29.13	29.28	29.50	30.53	29.53	
50 % NPK +4g/plant humic acid	29.80	27.20	27.99	27.65	29.20	28.50	27.76	28.30	
Mean (B)	29.01	28.63	28.83	28.18	29.89	29.28	29.43	-----	
LSD at 0.05 level	A= 0.63	B= 0.95			AB=1.64				
2014/2015 season									
100 % NPK (Control)	26.20	29.70	28.86	30.10	28.06	30.53	28.20	28.81	
75 % NPK +2g/plant humic acid	27.83	31.13	28.86	29.93	28.50	28.76	29.06	29.15	
50 % NPK +4g/plant humic acid	27.60	29.92	29.96	27.60	29.86	29.13	27.63	28.81	
Mean (B)	27.21	30.25	29.23	29.21	28.81	29.47	28.30	-----	
LSD at 0.05 level	A= NS	B= 0.92			AB=1.60				

Table (3): Effect of different fertilization levels, nanopartic and their interaction on branch number / plant of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Fertilization levels	Control		Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)
	0	60	20	40	60	20	40	60	
			2013/2014 season						
100 % NPK (Control)	6.70	8.54	6.25	6.51	8.54	8.66	6.81	7.71	7.31
75 % NPK +2g/plant humic acid	7.26	7.00	6.61	7.00	7.00	6.94	7.41	8.40	7.23
50 % NPK +4g/plant humic acid	7.30	7.00	6.53	7.75	7.00	6.86	7.56	6.37	7.05
Mean (B)	7.08	7.51	6.46	7.08	7.51	7.49	7.26	7.49	-----
LSD at 0.05 level	A= 0.16		B= 0.44			AB= 0.77			
	2014/2015 season								
100 % NPK (Control)	7.16	7.96	6.70	7.53	7.96	8.26	7.09	7.70	7.48
75 % NPK +2g/plant humic acid	7.20	8.40	7.52	6.40	8.40	7.43	6.70	8.00	7.40
50 % NPK +4g/plant humic acid	7.50	7.90	7.30	7.46	7.90	6.36	8.40	6.93	7.37
Mean (B)	7.28	8.08	7.17	7.13	8.08	7.35	7.39	7.54	-----
LSD at 0.05 level	A= NS		B= 0.59			AB=1.02			



Table (4): Effect of different fertilization levels, nanoparticles and their interaction on number of umbels/ plant of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Fertilization levels	Nano particles	Control			Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)	
		0			20	40	60	20	40	60		
		2013/2014 season										
100 % NPK (Control)		34.73	57.76	59.05	44.66	56.16	53.93	51.53	51.12			
75 % NPK +2g/plant humic acid		44.73	52.33	55.11	53.86	50.48	57.02	55.95	52.78			
50 % NPK +4g/plant humic acid		56.08	47.93	39.82	34.33	42.03	46.60	38.70	43.64			
Mean (B)		45.18	52.67	51.33	44.28	49.56	52.51	48.72	-----			
LSD at 0.05 level		A = 2.93			B = 3.79			AB = 6.56				
		2014/2015 season										
100 % NPK (Control)		47.80	51.90	68.66	45.66	57.60	53.30	54.13	54.15			
75 % NPK +2g/plant humic acid		45.00	59.83	54.53	65.76	48.20	46.03	51.66	53.00			
50 % NPK +4g/plant humic acid		42.58	47.53	53.66	47.03	55.60	55.06	48.43	49.98			
Mean (B)		45.12	53.08	58.95	52.82	53.80	51.46	51.41	-----			
LSD at 0.05 level		A = 3.23			B = 5.15			AB = 8.92				

These results may be attributed to nanoparticles effect in a number of crops in enhancing germination and seedling growth, physiological activities, gene expression and protein level indicating their potential use in crop improvement (Kole, *et al.*, 2013).

The data in Tables (2 to 4) revealed that the interaction between fertilization treatments and foliar spraying of nanoparticle showed significant effects on plant height and number of both branches and umbels in both seasons. In the first season, the tallest plants were resulted from plants received 100% NPK 20 nano graphite or 75 % NPK +2g/l humic acid as well as 60 mg/l nano graphite or 100 % NPK + 60 mg/l nano graphite without significant differences among themselves. In the second season, the significantly tallest plants resulted from either adding 75 % NPK +2g/l humic acid and 20 mg/l silica NPs or 100 % NPK with 60 mg/l silica NPs or 40 mg/l nano graphite. On the opposite, the significantly shortest plants were resulted from adding 50 % NPK+4mg/l humic acid and either 20 or 60 mg/l silica NPs in both seasons, respectively.

Regarding the number of branches, the significantly highest values were resulted in the first season from plants received 100 % NPK with 20 mg/l graphite NPs or 100 % NPK with 60 mg/l silica NPs or 75 % NPK +2g/l humic acid with 60 mg/l graphite NPs without significant differences among themselves. The highest umbels number/ plant was observed at 100 % NPK with 40 mg/l silica NPs in the two seasons, respectively. On the other hand, the significantly least values resulted from 100% NPK with 60 mg/l silica NPs in both seasons, respectively. The positive morphological effects of nanomaterials included enhancing germination percentage and rate; length of root and shoot, and their ratio; and vegetative biomass. (Amin *et al.*, 1999).

## **2- Dry weight, yield of seeds/ plant and 1000 seeds weight:**

Data presented in Tables (5 to 7) revealed that the significantly highest values of dry weight and seed yield resulted from NPK at full dose (control) in both seasons. On the other hand, using 50 % NPK+4g/plant humic acid recorded the significantly least values of plant dry weight and seed yield for the two seasons, respectively. Also, data cleared that the highest values of seed index (1000 seeds weight) resulted from 50 % NPK with 4g/plant humic acid in the first season and 100 % NPK in the second one.

The superiority of NPK in plant dry weight, seeds yield/ plant and 1000 seeds weight/ plant may be due to that chemical fertilizers could enhance plant growth due to the role of nitrogen in nucleic acids and protein synthesis, and phosphorus as an essential component of the energy compounds (ATP and ADP) and phosphoprotein, in addition to the role of potassium as an activator of many enzymes (Helgi and Rolfe, 2005).

Concerning the nanoparticles treatments, data showed that the significantly heaviest dry weight of plants, seeds yield/ plant and 1000 seeds weight resulted from applying 20 mg/l nano graphite in the 1<sup>st</sup> season and 40 mg/l graphite or 40 mg/l nano silica in the 2<sup>nd</sup> season, respectively. However, the least values of dry weight, seed yield/plant and 1000 seeds weight resulted from control plants in the first and second seasons,

This result may be attributed to the important role of silicon to be absorbed into plant to increase disease and stress resistance by promoting the physiological activity and growth of the plant. (Agrawal and Rathore, 2014).

The significantly highest values of dry weight resulted from plants received 50 % NPK +4g /plant humic acid with 20 or 40 mg/l graphite NPs in the two seasons. The highest values of seed yield/ plant were recorded from 100 % NPK and 40 mg/l nano graphite, while the highest values of 1000 seeds weight resulted from 75 % NPK +2g/plant humic acid + 60 mg/l silica NPs in both seasons. However, the least values of dry weight and yield of seeds/ plant resulted from plants received 50 % NPK + humic acid

Table (5): Effect of different fertilization levels, nanoparticles and their interaction on dry weight/plant(g) of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Nano particles Fertilization levels	Control			Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)
	0	20	40	60	20	40	60	20	40	
<b>2013/2014 season</b>										
100 % NPK (Control)	6.68	9.23	8.05	8.58	7.81	10.08	11.23	8.81		
75 % NPK +2g/plant humic acid	7.21	6.98	7.14	8.80	9.72	7.86	7.54	7.89		
50 % NPK +4g/plant humic acid	4.90	8.60	5.96	6.31	11.34	8.17	6.08	7.34		
Mean (B)	6.26	8.27	7.05	7.90	9.26	8.70	8.28	-----		
LSD at 0.05 level	A= 0.07			B= 0.59			AB=1.03			
<b>2014/2015 season</b>										
100 % NPK (Control)	5.82	6.67	9.34	8.19	8.32	11.37	7.83	8.22		
75 % NPK +2g/plant humic acid	5.93	8.74	8.27	8.69	6.24	7.47	9.16	7.78		
50 % NPK +4g/plant humic acid	4.44	8.22	9.04	6.10	6.88	11.93	6.84	7.63		
Mean (B)	5.39	7.87	8.88	7.66	7.14	10.26	7.94	-----		
LSD at 0.05 level	A= 0.42			B= 0.42			AB=0.73			

Table (6): Effect of different fertilization, nanoparticles and their interaction on yield of seeds/ plant (g) of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Nano particles Fertilization levels	Control			silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)	
	0			40			60				
	20	40	60	20	40	60	20	40	60		
2013/2014 season											
100 % NPK (Control)	4.19	6.12	5.38	5.40	5.09	6.19	4.88	5.32			
75 % NPK +2g/plant humic acid	3.63	4.25	4.72	5.27	5.69	4.66	4.91	4.73			
50 % NPK +4g/plant humic acid	2.88	4.17	4.53	4.16	5.34	4.52	3.78	4.20			
Mean (B)	3.57	4.85	4.87	4.94	5.37	5.12	4.52	-----			
LSD at 0.05 level	A= 0.15			B= 0.13			AB=0.24				
2014/2015 season											
100 % NPK (Control)	3.83	4.45	6.06	5.57	5.79	6.71	5.46	5.41			
75 % NPK +2g/plant humic acid	3.37	5.66	6.06	4.35	5.49	5.44	5.43	5.11			
50 % NPK +4g/plant humic acid	2.73	4.75	5.30	4.73	3.81	4.22	3.71	4.18			
Mean (B)	3.31	4.95	5.81	4.88	5.03	5.46	4.86	-----			
LSD at 0.05 level	A= 0.03			B= 0.11			AB=0.20				

*Effect of nano fertilization, chemical and humic acid on the .....*

Table (7): Effect of different fertilization levels, nanoparticles and their interaction between them on 1000 seeds weight (g) of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Fertilization levels	Nano particles		Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)
	Control	0	20	40	60	20	40	60	
	2013/2014 season								
100 % NPK (Control)	5.73	5.43	5.00	5.40	5.53	5.30	5.33	5.39	
75 % NPK +2g/plant humic acid	4.33	4.90	4.76	5.13	5.86	4.56	4.96	4.93	
50 % NPK +4g/plant humic acid	5.86	5.30	5.13	5.30	5.80	5.86	4.93	5.45	
Mean (B)	5.31	5.21	4.96	5.27	5.73	5.24	5.07	-----	
LSD at 0.05 level	A= 0.32		B= 0.58			AB=1.01			
2014/2015 season									
100 % NPK (Control)	5.23	4.93	6.30	5.90	6.23	4.70	5.03	5.47	
75 % NPK +2g/plant humic acid	5.03	4.40	4.90	6.36	5.56	5.80	3.96	5.14	
50 % NPK +4g/plant humic acid	5.50	5.56	5.80	4.13	3.83	5.60	6.00	5.20	
Mean (B)	5.25	4.96	5.66	5.46	5.21	5.36	5.00	-----	
LSD at 0.05 level	A= NS		B= NS			AB=1.25			

without nanopractices. On the contrary, the least values of 1000 seeds weight were obtained from 75 % NPK +2g/plant humic acid without nanopractices and 50 % NPK +4g/plant humic acid with 20 mg/l nano graphite.

These results may be due to the increment in soil fertility by NPK fertilizer as well as the benefits of humic acid, as improve soil structure and change physical properties of the soil (Chen and Avid, 1990). Also, nanoparticles have enhanced reactivity due to enhanced solubility, greater proportion of surface atoms relative to the interior of a structure, unique magnetic/optical properties, electronic states, and catalytic reactivity that differ from equivalent bulk materials (Agrawal and Rathore, 2014).

### **3- Chlorophyll a,b and carotenoides**

Data presented in Tables (8 to10) showed that the highest values of chlorophyll a, b and carotenoides resulted from using 75 % NPK+ 2g/plant humic acid or 50 % NPK + 4g/plant humic acid in leaf tissues in both seasons. On the opposite, the significantly least values resulted from applying 100 % NPK (control) in both seasons. These results are similar to those of Befrozfar *et al.* (2013) on *Ocimum bacilicum*.

Concerning the nanoparticles treatments, generally, data showed that, the significantly highest values of chlorophyll a, b and carotenoides resulted from applying 40 mg/ l silica NPs and 60 mg/ l graphite NPs in the 1<sup>st</sup> and 2<sup>nd</sup>, respectively.

Data illustrated also that the highest values of chlorophyll (a) resulted from using 50 % NPK +4g/plant humic acid+ 40 mg/l silica NPs or 75 % NPK +2g/plant humic acid + 40 mg/l graphite NPs in the first season. However, treatments of 50 % NPK + 4g/plant humic acid with 60 mg/l graphite NPs or 50 % NPK +4g /plant humic acid with 40 mg/l graphite NPs increased the chlorophyll (a) in the second season. The least values of chlorophyll (a) in the first and second seasons resulted from using 100 % NPK + 40 mg/l graphite NPs and 75 % NPK + 2g/plant humic acid with 40 mg/l silica NPs in the first and second seasons, respectively. Data cleared also that the best results of chlorophyll (b) were obtained from 75 % NPK +2g/plant humic acid with 60 mg/ l graphite NPs and 75 % NPK +2g/plant humic acid with 40 mg/ l graphite NPs in the two seasons, respectively. On the contrary, the least values were obtained from using 50 % NPK + humic acid with 40 mg/ l graphite NPs and 50 % NPK +4g/plant humic acid with 40 mg/ l silica NPs in the two seasons, respectively. Also, data revealed that, carotenoides showed nearly a similar trend as chlorophyll a, the highest values resulted from 50 % NPK +4g /plant humic acid with 40 mg/ l silica NPs and 60 mg/ l graphite NPs as compared to 100 % NPK with 40 mg/l graphite NPs and 50 % NPK +4g/plant humic acid with 20 mg/l graphite NPs in the 1<sup>st</sup> and 2<sup>nd</sup> seasons, respectively.

The enhancing effect on chlorophyll a due to applying nanoparticles may be due to that silica nanoparticles increase turgid pressure and plant size by improving water use efficiency and leaf relative water content (Rawson *et al.*, 1988)

This result is similar to those of El-Fouly *et al.* (2014) on *Cordyline terminalis*

### **4- Nitrogen, phosphorous and potassium percentage:**

Data presented in Tables (11 to 13) showed that the applying 100 % NPK gave the highest values of N and K % in the second season, while potassium and phosphorus gave the highest values with 75% NPK +2g/plant humic acid in both seasons.

This result is in conformity to Befrozfar *et al.* (2013) on *Ocimum bacilicum*.

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Concerning the nano particles treatments, data showed that the significantly highest values of nitrogen (%) resulted from applying the treatments of 20

Table (8): Effect of different fertilization levels, nanoparticles and their interaction on chlorophyll a (mg/gm FW) in herb of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Fertilization levels	Nano particles		Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)
	Control	0	20	40	60	20	40	60	
	2013/2014 season								
100 % NPK (Control)	2.60		2.92	3.47	3.61	3.23	2.66	3.31	3.11
75 % NPK +2g/plant humic acid	2.95		3.62	3.37	3.45	3.54	4.05	3.65	3.52
50 % NPK +4g/plant humic acid	3.60		3.15	4.30	2.81	3.64	2.69	3.67	3.41
Mean (B)	3.05		3.23	3.71	3.29	3.47	3.13	3.54	-----
LSD at 0.05 level	A = 0.18		B = 0.37			AB = 0.65			
2014/2015 season									
100 % NPK (Control)	2.58		3.08	3.07	3.48	3.59	3.40	3.37	3.23
75 % NPK +2g/plant humic acid	4.61		3.14	2.91	3.28	3.12	3.52	3.87	3.49
50 % NPK +4g/plant humic acid	4.23		3.19	3.55	3.35	3.29	3.96	4.74	3.76
Mean (B)	3.81		3.14	3.18	3.37	3.33	3.63	3.99	-----
LSD at 0.05 level	A = 0.40		B = 0.40			AB = 0.69			



Table (9): Effect of different fertilization levels, nanoparticles and their interaction on chlorophyll b (mg/gm FW) in herb of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Fertilization levels	Nano particles		Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)
	Control	0	20	40	60	20	40	60	
	2013/2014 season								
100 % NPK (Control)	1.30	1.04	1.72	1.51	1.65	1.82	1.33	1.48	
75 % NPK +2g/plant humic acid	1.17	1.48	1.47	1.63	1.53	2.06	2.10	1.63	
50 % NPK +4g/plant humic acid	1.57	1.18	2.01	1.09	1.81	0.86	1.30	1.40	
Mean (B)	1.35	1.23	1.74	1.41	1.67	1.58	1.58	-----	
LSD at 0.05 level	A= NS			B= 0.38			AB=0.67		
2014/2015 season									
100 % NPK (Control)	1.28	1.37	1.25	1.19	1.53	1.53	1.34	1.36	
75 % NPK +2g/plant humic acid	1.99	1.36	1.39	1.26	1.51	2.01	1.88	1.63	
50 % NPK +4g/plant humic acid	1.83	1.39	1.16	1.45	1.49	1.72	1.99	1.58	
Mean (B)	1.70	1.37	1.27	1.30	1.51	1.75	1.74	-----	
LSD at 0.05 level	A= 0.12			B= 0.44			AB=0.76		

Table (10): Effect of different fertilization levels, nanoparticle and their interaction on **carotenoides** (mg/gm FW) in herb of *Cuminum cyminum* L during 2013/2014 and 2014/2015 seasons

Nano particles Fertilization levels	Control		Silicon NPs (mg/l)			graphite NPs (mg/l)			Mean (A)				
	0	20	40		60		20			40		60	
			20	40	60	20	40	60					
2013/2014 season													
100 % NPK (Control)	1.02	1.30	1.36	1.32	1.32	1.24	1.09	1.32	1.23				
75 % NPK +2g/plant humic acid	1.26	1.48	1.36	1.37	1.41	1.49	1.31	1.38					
50 % NPK +4g/plant humic acid	1.40	1.31	1.50	1.13	1.28	1.15	1.44	1.32					
Mean (B)	1.23	1.36	1.41	1.27	1.31	1.24	1.35	-----					
LSD at 0.05 level	A= 0.13		B= NS			AB=0.31							
2014/2015 season													
100 % NPK (Control)	0.94	1.18	1.20	1.32	1.46	1.36	1.27	1.25					
75 % NPK +2g/plant humic acid	1.85	1.10	1.12	1.31	1.09	1.37	1.49	1.33					
50 % NPK +4g/plant humic acid	1.58	1.32	1.28	1.29	1.07	1.49	1.95	1.43					
Mean (B)	1.46	1.20	1.20	1.31	1.21	1.41	1.57	-----					
LSD at 0.05 level	A= 0.16		B= 0.16			AB=0.28							

*Effect of nano fertilization, chemical and humic acid on the .....*

Table (11): Effect of different fertilization levels, nanoparticles and their interaction on nitrogen (%) in herb of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Fertilization levels	Nano particles		Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)
	Control	0	20		60	20		60	
			40	60	40	60			
2013/2014 season									
100 % NPK (Control)	1.53		2.05	1.55	1.60	1.78	1.57	1.72	1.69
75 % NPK +2g/plant humic acid	1.45		1.71	1.62	1.82	2.17	1.60	1.48	1.69
50 % NPK +4g/plant humic acid	1.63		2.10	1.72	1.72	2.01	1.34	1.55	1.72
Mean (B)	1.54		1.95	1.63	1.71	1.99	1.50	1.58	-----
LSD at 0.05 level	A= NS		B=0.04			AB=0.07			
2014/2015 season									
100 % NPK (Control)	1.57		2.19	1.61	1.75	1.87	1.58	1.50	1.72
75 % NPK +2g/plant humic acid	1.62		1.73	1.51	1.52	1.92	1.75	1.57	1.66
50 % NPK +4g/plant humic acid	1.87		1.73	1.56	1.74	1.92	1.55	1.51	1.70
Mean (B)	1.69		1.88	1.56	1.67	1.91	1.63	1.52	-----
LSD at 0.05 level	A= 0.02		B= 0.04			AB=0.07			

*Effect of nano fertilization, chemical and humic acid on the .....*

Table (12): Effect of different fertilization levels, nanoparticles and their interaction on phosphorus (%) in herb of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Fertilization levels	Nano particles		Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)
	Control	0	20	40	60	20	40	60	
	2013/2014 season								
100 % NPK (Control)	0.17	0.17	0.50	0.22	0.31	0.18	0.22	0.10	0.24
75 % NPK +2g/plant humic acid	0.23	0.23	0.22	0.31	0.41	0.33	0.35	0.33	0.31
50 % NPK +4g/plant humic acid	0.28	0.28	0.34	0.23	0.24	0.25	0.29	0.40	0.29
Mean (B)	0.22	0.22	0.35	0.25	0.32	0.25	0.28	0.27	-----
LSD at 0.05 level	A= NS		B= 0.07			AB=0.13			
2014/2015 season									
100 % NPK (Control)	0.18	0.18	0.47	0.23	0.28	0.17	0.24	0.35	0.27
75 % NPK +2g/plant humic acid	0.22	0.22	0.23	0.33	0.39	0.33	0.34	0.39	0.32
50 % NPK +4g/plant humic acid	0.29	0.29	0.37	0.25	0.28	0.26	0.29	0.40	0.30
Mean (B)	0.23	0.23	0.36	0.27	0.31	0.25	0.29	0.38	-----
LSD at 0.05 level	A= 0.01		B= 0.01			AB=0.02			

*Effect of nano fertilization, chemical and humic acid on the .....*

Table (13): Effect of different fertilization levels, nano particles and their interaction on potassium (%) in herb of *Cuminum cyminum* L. during 2013/2014 and 2014/2015 seasons

Fertilization levels	Nano particles		Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)
	Control	0	20		40		60		
			20	40	20	40	60		
2013/2014 season									
100 % NPK (Control)	2.56	2.63	2.35	2.45	2.63	2.61	2.83	2.58	
75 % NPK +2g/plant humic acid	2.70	2.68	2.63	2.53	2.80	2.52	2.77	2.66	
50 % NPK +4g/plant humic acid	2.81	2.60	2.68	2.58	2.65	2.44	2.52	2.61	
Mean (B)	2.69	2.63	2.55	2.52	2.69	2.52	2.71	-----	
LSD at 0.05 level	A=0.06		B= 0.07			AB=0.12			
2014/2015 season									
100 % NPK (Control)	2.51	2.86	2.52	2.49	2.86	2.75	3.12	2.73	
75 % NPK +2g/plant humic acid	2.53	2.59	2.54	2.55	2.71	2.54	2.97	2.63	
50 % NPK +4g/plant humic acid	2.91	2.43	2.54	2.73	2.66	2.38	2.48	2.59	
Mean (B)	2.65	2.63	2.53	2.59	2.74	2.56	2.85	-----	
LSD at 0.05 level	A= 0.04		B= 0.07			AB=0.12			

## **Effect of nano fertilization, chemical and humic acid on the .....**

mg/l graphite NPs. The highest values of phosphorus were registered from using 20 mg/l silicon NPs while potassium showed the highest percent for 60 mg/l graphite NPs in the two seasons. On the other hand, the least values of nitrogen resulted from 40 mg/l and 60 mg/l graphite NPs in both seasons, respectively. The highest Phosphorus values were observed for 40 mg/l silica NPs or 20 mg/l graphite NPs and 20 mg/l graphite NPs in the 1<sup>st</sup> and 2<sup>nd</sup> seasons, respectively. However, the least values resulted from 60 mg/l silica NPs or 40 mg/l graphite NPs in the first season and 40 mg/l silica NPs in the second one. These results are in agreement with those of El Kereti *et al.* (2013) on *Ocimum bacilicum*.

For the interaction between different fertilization levels and nanoparticles, the highest values of N (%) resulted from plants received 75 % NPK + humic acid + 20 mg/l graphite NPs and 100 % NPK + 20 mg/l silica NPs in the 1<sup>st</sup> and 2<sup>nd</sup> seasons, respectively. The least values resulted from the treatments of 50 % NPK + humic acid with 40 mg/l graphite NPs and 100 % NPK with 60 mg/l graphite NPs. Regarding phosphorous (%), the highest values were attained from the plants received 100 % NPK with 20 mg/l silica NPs in both seasons, while, the least values were obtained from using 100 % NPK with 60 mg/l graphite NPs and 100 % NPK with 20 mg/l graphite NPs. Concerning potassium percentage, the highest values were obtained from using 100 % NPK with 60 mg/l graphite NPs in both seasons. On the other hand, the least values were recorded from using 75 % NPK +2g/plant humic acid with 60 mg/l silica NPs and 50 % NPK +4g/plant humic acid with 40 mg/l graphite NPs.

These results are in conformity to those of Gomaa and Youssef (2008) on caraway.

### **5- Oil percentage:**

Data presented in Table (14) showed that using full dose of NPK gave the significantly highest values of oil percent. The least value were obtained from using 50 % NPK + humic acid in both seasons.

These results are in agreement with those of Juarez R. *et al.* (2011) on *Thymus vulgaris* L. and Nasirolesl and Safaridolatabad (2014) on dill.

Concerning nanoparticles treatments, data pointed out the highest oil percentage resulted from the treatments of 60 mg/l nano graphite in both seasons. On the other side, the treatment of 20 mg/l nano graphite and 60 mg/l nano silica recorded the significantly least oil percent. The augmentation of oil percentage from adding nanoparticles treatments may be due to that in the case of foliar spray, this would result in a high photosynthesis rate and consequently more production of carbohydrate precursors for essential oil synthesis.

For the interaction between different fertilization levels and nanoparticles treatments data showed that, the significantly highest oil % resulted from treated plants with 100 % NPK + 60 mg/l nano graphite in both seasons. The least value of oil % resulted from the treatment of 100 % NPK + 20 mg/l nano graphite and 50 % NPK + humic acid with 60 mg/l nano graphite in the first and second season, respectively.

These results are in harmony with those El- Kereti *et al.* (2013) on sweet basil.

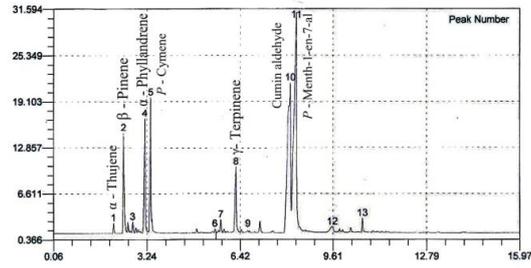
### **Essential oil constituents:**

Components in volatile oil of different treatments were identified. Data indicated that, the major components which were identified were *p*-mentha-1-en-7-al as recorded 41.47 % from the treatment of 75 % NPK +2g/plant humic acid with 40 mg/l silica NPs followed by 50 % NPK + 4g/plant humic acid and 60 mg/l graphite NPs and cumin aldehyde as recorded 36.67 % from the treatment 100 % NPK with 20 mg/l graphite NPs. On the opposite, the least values were obtained from the treatment of 100 % NPK with 20 mg/l silicon NPs as recorded 31.28 %, 23.82 %, respectively. The other components which were identified in the oil were  $\alpha$ - thujene,  $\beta$ -

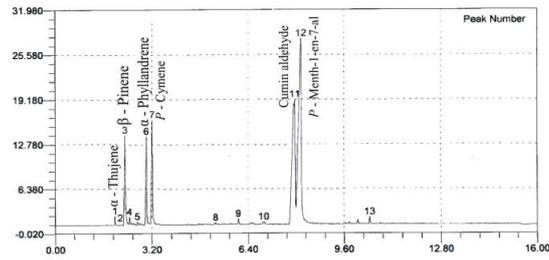
pinene,  $\alpha$ - phyllandrene, *P*- cymene and  $\gamma$ - terpinene (as shown in Fig. 3: a,b,c,d and f ). These results are in harmony with those of Beis *et*

Table (14): Effect of mineral fertilization levels, nanoparticles and their interaction on essential oil (%) of *Cuminum cyminum* L. seeds during 2013/2014 and 2014/2015 seasons

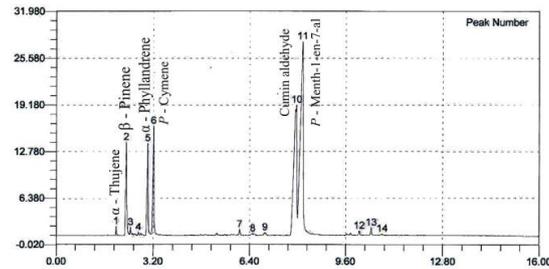
Fertilization levels	Nano particles	Control			Silicon NPs (mg/l)			Graphite NPs (mg/l)			Mean (A)			
		0			20			40				60		
		2013/2014 season												
100 % NPK (Control)		4.53	5.53	5.26	5.33	4.13	4.83	5.66	5.04					
75 % NPK +2g/plant humic acid		4.23	5.10	4.43	5.16	5.16	4.86	5.16	4.87					
50 % NPK +4g/plant humic acid		3.96	5.16	5.10	4.66	4.66	4.30	5.10	4.70					
Mean (B)		4.24	5.26	4.93	5.05	4.65	4.66	5.31	-----					
LSD at 0.05 level		A= 0.17			B= 0.22			AB=0.39						
		2014/2015 season												
100 % NPK (Control)		4.36	5.10	4.80	4.63	5.16	4.36	5.83	4.89					
75 % NPK +2g/plant humic acid		4.83	4.93	4.86	4.46	4.63	4.83	5.26	4.83					
50 % NPK +4g/plant humic acid		3.66	4.40	5.06	4.60	4.26	4.66	3.83	4.35					
Mean (B)		4.28	4.81	4.91	4.56	4.68	4.62	4.97	-----					
LSD at 0.05 level		A= 0.22			B= 0.22			AB=0.39						



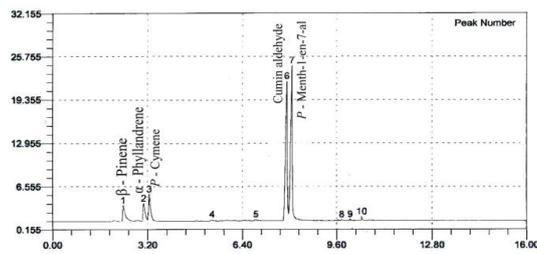
A) 100 % NPK (control) without nano.



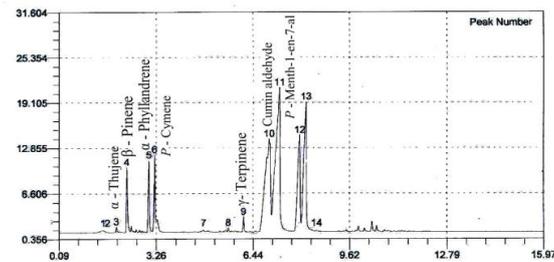
B) 75 % NPK + humic acid + 40 mg/l nano silicon.



C) 50 % NPK + humic acid + 60 mg/l nano graphite.



D) 100 % NPK + 20 mg/l nano graphite.



F) 100 % NPK + 20 mg/l nano silicon

**Fig (3): Effect of different fertilization kinds and levels on cumin essential oil composition.**

al. (2000) and Iacobellis *et al.* (2005) who reported that *p*-mentha-1,4dien-7al as the major component with a concentration 27.4 % whereas cumin aldehyde secured second rank with a concentration of 16.1 %.

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## تأثير معاملات التسميد بالنانو والتسميد الكيماوى و حمض الهيوميك على النمو الخضرى و التركيب الكيماوى و محصول الزيت فى نبات الكمون

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### الملخص العربى

أجريت هذه التجربة فى مزرعة خاصة بالقرب من المحلة الكبرى (محافظة الغربية) أثناء موسمى 2013-2014 ،  
2014-2015 لدراسة تأثير مستويات تسميدية مختلفة بالإضافة للتسميد بالنانو سليكون و النانو جرافيت على  
النمو الخضرى و التركيب الكيماوى و محصول الزيت و تركيبه لنبات الكمون. و قد تم تقدير حجم جزيئات النانو  
باستخدام الميكروسكوب الالكترونى و تم رش النبات بثلاث تركيزات 20، 40، 60 ملليجرام/ لتر من كلا المادتين  
مرتين. و قد أوضحت النتائج انه للحصول على أفضل نمو خضرى ( طول النبات، عدد الأفرع و النورات) تمت  
معاملة النباتات ب75 % من التسميد الكيماوى الموصى به +2 جم/نبات حمض الهيوميك مع الرش ب60  
ملليجرام/ لتر نانو جرافيت، و تم تحقيق اعلى قيمة لكلورفيل أ، ب من معاملة النباتات ب75 % من التسميد  
الكيماوى الموصى به + 2 جم/نبات حمض الهيوميك مع الرش ب40 ملليجرام/ لتر نانو جرافيت، بينما معاملة  
النباتات بالتسميد الكيماوى الموصى به مع الرش ب20 ملليجرام/ لتر نانو سيلكا اعطت اعلى نسبة مئوية لكل من  
النيتروجين و الفوسفور و للحصول على اعلى نسبة مئوية للزيت و البوتاسيوم يجب معاملة النباتات بالتسميد  
الكيماوى الموصى به مع الرش ب60 ملليجرام/ لتر نانو جرافيت و حققت المعاملة ب75 % من التسميد الكيماوى  
الموصى به + 2 جم/نبات مع الرش ب40 ملليجرام/ لتر نانو سيلكا اعلى نسبة مئوية من مركب بيتا- ميثا-1، 7  
اول.

تبعاً للنتائج السابقة نوصى بالآتى:

- 1- للحصول على أفضل نمو خضرى تتم معاملة النباتات ب75 % من التسميد الكيماوى الموصى به + 2  
جم/نبات حمض الهيوميك مع الرش ب60 ملليجرام/ لتر نانو جرافيت.
- 2- للحصول على أعلى محصول زيت تتم معاملة النباتات بالتسميد الكيماوى الموصى به مع الرش ب60  
ملليجرام/ لتر نانو جرافيت.