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Effect of nano iron oxide application on iron availability in calcareous vertisols

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Abstract

Incubation studies on dynamics of applied nano iron oxides in calcareous Vertisol was conducted for a period of 60 days at department of Soil Science and Agricultural Chemistry, University of Agricultural Sciences, Dharwad. The results revealed that, DTPA iron in soil decreased with advancement in period of incubation. Highest DTPA extractable iron (6.87, 5.97, 4.89 and 4.74 mg kg⁻¹ at 15, 30, 45 and 60 DAI, respectively) and water soluble iron (1.59, 1.81, 1.92 and 1.72 mg kg⁻¹ at 15, 30, 45 and 60 DAI, respectively) was recorded in the treatment receiving nano Fe₂O₃ @ 2.5 mg kg⁻¹ mixed with FYM. The water soluble iron increased in all the treatments up to 45 DAI and thereafter, decreased. The treatments which received iron oxide formulations chelated with FYM recorded higher DTPA extractable and water soluble iron than their respective unchelated ones at all the concentration levels. Between the sizes, the nanoparticles having the size < 50 nm were found better than the size range of particles between 50 - 100 nm in maintaining soil DTPA iron and water soluble iron status at all concentration ranges throughout the incubation period.

Keywords: Nano iron oxide, incubation, chelation, DTPA extractable iron, water soluble iron

Introduction

Iron is an important element and ranks fourth in abundance in the earth's crust after oxygen, silicon and aluminium. Iron plays many essential roles in plant growth and development including thylakoid synthesis, chlorophyll synthesis, chloroplast development, contributes to RNA synthesis and improves the performance of photosystems (Malakouti and Tehrani, 2005)^[15]. Through its ability to accept and donate electrons, iron plays major roles in the electron transport chains of photosynthesis, respiration and energy transfer in the plant system. Iron acts as a co-factor for approximately 140 enzymes that catalyze important biochemical reactions (Brittenham, 1994)^[15]. Lack of iron causes young leaves to turn yellow and reduction in photosynthetic activity (Briat *et al.*, 2007)^[14].

Calcareous soils (soils having more than 5 per cent CaCO₃) cover over 30 per cent of the earth's land surface. Nutrient management in calcareous soils different from that of non-calcareous soils because of the effect of pH on soil nutrient availability and chemical reactions that affect the loss or fixation of almost all nutrients. Iron deficiency is common in calcareous soils that have a high CaCO₃ due to reduced solubility of iron at alkaline pH values. Calcareous soils may contain high levels of total Fe, but in forms unavailable to plants. The plants grown in calcareous soils may exhibit chlorotic symptoms. Thus, this disorder in calcareous soils is not always attributable to iron content but due to its non availability, this condition is known as lime induced iron chlorosis. Iron deficiency in extreme cases may lead to complete crop failure.

With the rapid advancement of science, nanotechnology is being considered as a field that has potential to revolutionize agriculture production and food systems. Nanotechnology is gradually marching from the experimental stage to the stage of operational and practical (Baruah and Dutta, 2009)^[2]. Nano powders are mixture of particles with dimensions between 1 to 100 nm. With the introduction of high analysis fertilizers and excessive use of chemicals in the past century, we have ended up with polluted soils and underground waters. One of the most important applications of nanotechnology in agriculture is using nano fertilizers for plant nutrition. Altering micronutrients to become nano fertilizers may help to enhance the use efficiency of applied fertilizers besides quality and yield of the produce.

The use of nano fertilizer to control release of nutrients can be an effective step towards achieving sustainable agriculture and environment (Cui, 2006) [7].

The unusual properties of nanoparticles may result in altogether different behavior and environmental fate than their bulk counterparts. Nano particles, being small size (less than 100 nm) are having high surface area, high activity, better catalytic surface, rapid chemical reaction, highly dispersible and adsorb abundant water. Reduction of particle size results in increased number of particles per unit of weight and specific surface area of a fertilizer that should increase contact of fertilizer with plant leading to increase in nutrient uptake (Liscano *et al.*, 2000) [12]. Use of chelated forms which are stabilized by reaction of the metal salts with natural and synthetic complexes are the most important ways to protect iron from the precipitation at increased soil pH. Thus, nano fertilizers may increase the use efficiency of applied nutrients. In view of the above facts, an attempt was made to study the effect of soil application of nano iron on DTPA extractable iron, water soluble iron and total iron.

Material and Methods

Incubation experiment

Incubation studies on dynamics of applied nano iron oxides in calcareous Vertisol was conducted at Department of Soil Science and Agricultural Chemistry, College of Agriculture, University of Agricultural Sciences, Dharwad. One kg of air dried 2 mm sieved soil sample was kept in polythene bags of size 15 cm x 10 cm. The required amount of nano iron oxide powder was dissolved in 500 ml deionised water and dispersed by sonicator for 30 min to avoid aggregation of the particles. Then, the suspension was mixed with soil and chelation with FYM in the ratio of 1:100 has done as per treatment details and incubated at field capacity for 60 days. Water soluble iron, DTPA extractable iron and total iron were measured at 15, 30, 45 and 60 days after incubation.

Soil characteristics

A bulk soil sample was (0-15 cm) collected from MARS, Dharwad (F block, plot no. 136) and used for incubation experiment. A surface composite soil sample was collected processed and analyzed for initial soil chemical properties by employing standard methods. The soil was clay in texture and slightly alkaline in reaction (pH 7.6) with low electrical conductivity (0.17 dS m⁻¹). The organic carbon content was medium (5.80 g kg⁻¹) and calcareous in nature (CaCO₃ 10.5%) with low available nitrogen (210 kg N ha⁻¹), medium available phosphorus (41.60 kg P₂O₅ ha⁻¹) and high available potassium (485 kg K₂O ha⁻¹). The soil was sufficient in all micro nutrients except zinc (0.44 mg Kg⁻¹) and iron (4.13 mg Kg⁻¹). Total iron in the initial soil sample was 4.87% and water soluble iron was 0.92 mg Kg⁻¹. For estimating water soluble iron, soil was extracted with water (double distill) maintaining soil to water ratio of 1:5 and equilibrated for 2 hours by shaking on a mechanical shaker. Iron in the extract was determined by Atomic absorption Spectrophotometer. (Ma and Uren. 1995) [14]. Total iron in the incubated sample was estimated by hydrofluoric acid digestion method (Jackson, 1967) [11]. A 0.1 g finely ground sample was weighed in a platinum crucible and digested with HF - HClO₄ mixture. Sample was evaporated to dryness and gently boiled for 5 min by adding 6N HCl. The digested sample was filtered and made to known volume and total iron was estimated by AAS.

Characterization of iron oxide nanoparticles

Nano Fe₂O₃ (less than 50 nm) and Fe₃O₄ (50 - 100 nm) particles were purchased from Sigma Aldrich company. The morphology and particle size of nano iron oxides were confirmed by UV-visible spectrophotometer, field emission scanning electron microscope (FE-SEM) and X ray diffraction pattern of nanoparticles. Examination of the peaks and image obtained from SEM revealed that the particles are cubic, highly uniform in nature and the particle size of the nano Fe₂O₃ ranged from 20 - 45 nm whereas nano Fe₃O₄ ranged from 70 - 92 nm.

Statistical analysis

The statistical analysis and interpretation of data were done using the Fischer's method of analysis of variance technique as described by Gomez and Gomez (1984) [9]. Means were compared by Duncan's multiple range test and a 95% significance level ($p < 0.05$) was employed for all comparisons

Results and Discussion

DTPA extractable iron

The DTPA extractable iron differed significantly due to soil application of different size and level of nano iron formulations during the incubation period (Table 1). The DTPA iron found to increase in all the treatments compared to initial values. A significantly higher values 6.87, 5.97, 4.89 and 4.74 mg kg⁻¹ were recorded in the treatment which received nano Fe₂O₃ @ 2.5 mg kg⁻¹ having size < 50 nm mixed with FYM at 15, 30, 45 and 60 DAI, respectively. Lowest DTPA available iron observed in the control throughout the incubation period. Treatment receiving nano Fe₂O₃ @ 2.5 mg kg⁻¹ mixed with FYM having size range of 50 - 100 nm was on par with the superior treatment (T₆).

However, DTPA iron decreased at 30 45 and 60 DAI in all the treatments compared to 15 DAI. Decreased concentration of DTPA iron with the period of incubation might be due to the significant depressing effect of CaCO₃ and the high soil pH of calcareous soils as reported by Hellal *et al.* (2008) [10]. The decrease in DTPA iron with period of incubation was also reported by Sahrawat (2016) [17]. Many researchers reported less availability of iron in calcareous soils owing to its precipitation (Roosta, 2011 and Colla *et al.*, 2010) [16, 6]. Deb *et al.* (1990) [8] observed reduced recovery of applied Fe significantly with increase in period of incubation upto 60 days. The reduction in recovery was to the tune of 50 per cent at 20 DAI with the application of 10 and 20 ppm Fe and further at 60 DAI the available Fe content in soil reduced significantly.

Further, treatments which received iron oxide formulations chelated with FYM recorded higher DTPA extractable iron values than their respective unchelated ones at all the levels and periods of incubation. This may be due to the fact that addition of FYM chelates iron which releases iron slowly over an extended period. Further, FYM also prevents loss of iron from precipitation, oxidation and leaching (Sharma *et al.*, 2001) [18]. Between the sizes, the nanoparticles having the size < 50 nm were found better than the particles having size between 50 - 100 nm in maintaining soil DTPA iron status at all concentration ranges throughout the incubation period. This could be attributed to more specific area of smaller nanoparticles available for chelation with FYM (Liscano *et al.*, 2009) [12].

Water soluble iron

Water soluble iron was significantly influenced by application of different sized nano iron oxide formulations and period of incubation (Table 2). Highest water soluble iron (1.59 mg kg^{-1}) was recorded in the treatment (T_6) which received nano Fe_2O_3 @ 2.5 mg kg^{-1} having size $< 50 \text{ nm}$ mixed with FYM and lowest was observed in the control (0.56 mg kg^{-1}). Treatment T_7 (1.54) receiving nano Fe_2O_3 @ 2.5 mg kg^{-1} mixed with FYM was on par with the treatment T_6 .

Water soluble iron increased at 30 and 45 DAI in all the treatments compared to 15 DAI. Treatment receiving nano Fe_2O_3 ($< 50 \text{ nm}$) @ 2.5 mg kg^{-1} mixed with FYM recorded significantly higher values (1.81 and 1.92 mg kg^{-1} , respectively) and lower values (0.71 and 0.78 mg kg^{-1} , respectively) recorded in the control. The increase in water soluble iron with incubation time could be attributed to reductive Fe dissolution (Amrhein *et al.*, 1994) [1]. This increase in iron content could also due to slow oxidation of sparingly soluble iron into various insoluble higher oxidation states as reported by Hellal *et al.* (2008) [10].

Whereas at 60 DAI, the trend has changed, the amount of water soluble iron values decreased compared to 45 DAI. Treatment T_6 receiving Fe_2O_3 ($< 50 \text{ nm}$) @ 2.5 mg kg^{-1} with addition of FYM recorded significantly higher value (1.72 mg kg^{-1}) which was on par with treatment T_7 (1.65 mg kg^{-1}). The significant reduction of aqueous Fe concentrations after 45 days after incubation was probably as a result of FeCO_3 precipitation (Ma and Dong, 2004).

The status of water soluble iron in soil increased up to 45 DAI and thereafter decreased with the period of incubation. Further, treatments which received iron oxide formulations chelated with FYM recorded higher water soluble iron values than their respective unchelated ones at all the levels and periods of incubation. Between the sizes, the nanoparticles having the size $< 50 \text{ nm}$ were found better than the particles having size between $50 - 100 \text{ nm}$ in maintaining water soluble iron status at all concentration ranges throughout the incubation period.

Total iron

Total iron was not differed significantly due to application of different sized nano iron formulations during the incubation period (Table 3). However, highest total iron (5.02%) was observed in the Treatment (T_6) receiving nano Fe_2O_3 ($< 50 \text{ nm}$) @ 2.5 mg kg^{-1} mixed with FYM and lower value was observed in the control which received no fertilizer (4.89%) at 15 DAI. The same trend was followed throughout the incubation period. Total iron content in the soil increased upto 15 DAI and thereafter stabilized and almost reached a constant value without any definite trend. The fractions of iron such as water soluble, exchangeable acid soluble, lead displaceable, Mn oxide occluded, organically bound, Fe oxide occluded, amorphous and residual will be interchanging among themselves with the period of incubation as well as with the rates of application of fertilizers but the effect of incubation on total iron was non significant as it is the sum of the all the fractions.

Table 1: DTPA iron in soil as influenced by different size and levels of nano iron oxide formulations at different intervals of incubation

	Treatments	DTPA iron (mg kg^{-1})			
		15 DAI	30 DAI	45 DAI	60 DAI
T ₁	Control	4.61	3.66	3.43	3.32
T ₂	FeSO_4 @ 12.5 mg kg^{-1} ($25 \text{ kg FeSO}_4 \cdot 7\text{H}_2\text{O ha}^{-1}$)	5.01	4.52	4.19	4.11
T ₃	FeSO_4 @ 12.5 mg kg^{-1} mixed with FYM (1: 100)	5.42	4.96	4.39	4.23
T ₄	Nano Fe @ 2.5 mg kg^{-1} ($< 50 \text{ nm}$)	6.21	5.35	4.47	4.42
T ₅	Nano Fe @ 2.5 mg kg^{-1} ($50-100 \text{ nm}$)	6.05	5.13	4.38	4.35
T ₆	Nano Fe @ 2.5 mg kg^{-1} mixed with FYM (1: 100) ($< 50 \text{ nm}$)	6.87	5.97	4.89	4.74
T ₇	Nano Fe @ 2.5 mg kg^{-1} mixed with FYM (1: 100) ($50-100 \text{ nm}$)	6.52	5.60	4.70	4.51
T ₈	Nano Fe @ 1.25 mg kg^{-1} ($< 50 \text{ nm}$)	5.35	4.64	4.19	4.09
T ₉	Nano Fe @ 1.25 mg kg^{-1} ($50-100 \text{ nm}$)	5.32	4.55	4.11	4.01
T ₁₀	Nano Fe @ 1.25 mg kg^{-1} mixed with FYM (1: 100) ($< 50 \text{ nm}$)	5.78	5.16	4.32	4.27
T ₁₁	Nano Fe @ 1.25 mg kg^{-1} mixed with FYM (1: 100) ($50-100 \text{ nm}$)	5.66	4.87	4.21	4.11
T ₁₂	Nano Fe @ 0.5 mg kg^{-1} ($< 50 \text{ nm}$)	5.14	4.16	3.81	3.79
T ₁₃	Nano Fe @ 0.5 mg kg^{-1} ($50-100 \text{ nm}$)	5.06	4.11	3.72	3.65
T ₁₄	Nano Fe @ 0.5 mg kg^{-1} mixed with FYM (1: 100) ($< 50 \text{ nm}$)	5.27	4.43	4.01	3.91
T ₁₅	Nano Fe @ 0.5 mg kg^{-1} mixed with FYM (1: 100) ($50-100 \text{ nm}$)	5.21	4.41	3.93	3.85
T ₁₆	Nano Fe @ 0.25 mg kg^{-1} ($< 50 \text{ nm}$)	4.76	3.84	3.51	3.42
T ₁₇	Nano Fe @ 0.25 mg kg^{-1} ($50-100 \text{ nm}$)	4.71	3.73	3.48	3.36
T ₁₈	Nano Fe @ 0.25 mg kg^{-1} mixed with FYM (1: 100) ($< 50 \text{ nm}$)	4.92	4.22	3.65	3.52
T ₁₉	Nano Fe @ 0.25 mg kg^{-1} mixed with FYM (1: 100) ($50-100 \text{ nm}$)	4.85	3.93	3.61	3.49
S.Em. \pm		0.09	0.15	0.14	0.13

Note:

$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ @ 25 kg ha^{-1} ($12.5 \text{ mg FeSO}_4 \cdot 7\text{H}_2\text{O kg}^{-1}$ soil) is equivalent to 5 kg Fe ha^{-1} ($2.5 \text{ mg Fe kg}^{-1}$)

DAI –Days after incubation

Table 2: Water soluble iron in soil as influenced by different size and levels of nano iron oxide at different intervals of incubation

Treatments		Water soluble iron (mg kg ⁻¹)							
		15 DAI		30 DAI		45 DAI		60 DAI	
T ₁	Control	0.56	l	0.71	k	0.78	h	0.62	j
T ₂	FeSO ₄ @ 12.5 mg kg ⁻¹ (25 kg FeSO ₄ .7H ₂ O ha ⁻¹)	1.03	e-g	1.19	fg	1.26	ef	1.11	ef
T ₃	FeSO ₄ @ 12.5 mg kg ⁻¹ mixed with FYM (1: 100)	1.24	c	1.42	cd	1.53	d	1.35	c
T ₄	Nano Fe @ 2.5 mg kg ⁻¹ (< 50 nm)	1.41	b	1.59	b	1.67	c	1.49	b
T ₅	Nano Fe @ 2.5 mg kg ⁻¹ (50-100 nm)	1.37	b	1.54	bc	1.61	cd	1.42	bc
T ₆	Nano Fe @ 2.5 mg kg ⁻¹ mixed with FYM (1: 100) (< 50 nm)	1.59	a	1.81	a	1.92	a	1.72	a
T ₇	Nano Fe @ 2.5 mg kg ⁻¹ mixed with FYM (1: 100) (50-100 nm)	1.54	a	1.77	a	1.80	ab	1.65	a
T ₈	Nano Fe @ 1.25 mg kg ⁻¹ (< 50 nm)	1.10	de	1.28	ef	1.36	e	1.18	de
T ₉	Nano Fe @ 1.25 mg kg ⁻¹ (50-100 nm)	1.06	ef	1.23	fg	1.30	ef	1.13	ef
T ₁₀	Nano Fe @ 1.25 mg kg ⁻¹ mixed with FYM (1: 100) (< 50 nm)	1.22	c	1.43	cd	1.55	cd	1.34	c
T ₁₁	Nano Fe @ 1.25 mg kg ⁻¹ mixed with FYM (1: 100) (50-100 nm)	1.19	cd	1.40	de	1.50	d	1.30	cd
T ₁₂	Nano Fe @ 0.5 mg kg ⁻¹ (< 50 nm)	0.84	hi	1.02	hi	1.10	g	0.92	gh
T ₁₃	Nano Fe @ 0.5 mg kg ⁻¹ (50-100 nm)	0.79	ij	0.96	i	1.04	g	0.86	h
T ₁₄	Nano Fe @ 0.5 mg kg ⁻¹ mixed with FYM (1: 100) (< 50 nm)	0.96	fg	1.18	fg	1.29	ef	1.08	ef
T ₁₅	Nano Fe @ 0.5 mg kg ⁻¹ mixed with FYM (1: 100) (50-100 nm)	0.92	gh	1.12	gh	1.23	f	1.03	fg
T ₁₆	Nano Fe @ 0.25 mg kg ⁻¹ (< 50 nm)	0.63	kl	0.81	jk	0.90	h	0.70	ij
T ₁₇	Nano Fe @ 0.25 mg kg ⁻¹ (50-100 nm)	0.61	kl	0.79	k	0.87	h	0.67	j
T ₁₈	Nano Fe @ 0.25 mg kg ⁻¹ mixed with FYM (1: 100) (< 50 nm)	0.76	ij	0.98	i	1.11	g	0.88	h
T ₁₉	Nano Fe @ 0.25 mg kg ⁻¹ mixed with FYM (1: 100) (50-100 nm)	0.72	jk	0.93	ik	1.05	g	0.82	hi
S.Em. ±		0.04		0.04		0.04		0.05	

Note:

FeSO₄.7H₂O @ 25 kg ha⁻¹ (12.5 mg FeSO₄.7H₂O kg⁻¹ soil) is equivalent to 5 kg Fe ha⁻¹ (2.5 mg Fe kg⁻¹)

DAI –Days after incubation

Table 3: Total iron in soil as influenced by different levels of nano iron oxide at different intervals of incubation

Treatments		Total iron (%)							
		15 DAI		30 DAI		45 DAI		60 DAI	
T ₁	Control	4.89	a	4.87	a	4.88	a	4.87	a
T ₂	FeSO ₄ @ 12.5 mg kg ⁻¹ (25 kg FeSO ₄ .7H ₂ O ha ⁻¹)	4.95	a	4.93	a	4.94	a	4.93	a
T ₃	FeSO ₄ @ 12.5 mg kg ⁻¹ mixed with FYM (1: 100)	4.98	a	4.98	a	4.99	a	4.98	a
T ₄	Nano Fe @ 2.5 mg kg ⁻¹ (< 50 nm)	5.00	a	4.98	a	4.99	a	4.98	a
T ₅	Nano Fe @ 2.5 mg kg ⁻¹ (50-100 nm)	4.99	a	4.97	a	4.98	a	4.97	a
T ₆	Nano Fe @ 2.5 mg kg ⁻¹ mixed with FYM (1: 100) (< 50 nm)	5.02	a	5.01	a	5.02	a	5.03	a
T ₇	Nano Fe @ 2.5 mg kg ⁻¹ mixed with FYM (1: 100) (50-100 nm)	5.01	a	5.01	a	5.01	a	5.02	a
T ₈	Nano Fe @ 1.25 mg kg ⁻¹ (< 50 nm)	4.96	a	4.94	a	4.95	a	4.94	a
T ₉	Nano Fe @ 1.25 mg kg ⁻¹ (50-100 nm)	4.95	a	4.93	a	4.94	a	4.92	a
T ₁₀	Nano Fe @ 1.25 mg kg ⁻¹ mixed with FYM (1: 100) (< 50 nm)	4.98	a	4.97	a	4.97	a	4.98	a
T ₁₁	Nano Fe @ 1.25 mg kg ⁻¹ mixed with FYM (1: 100) (50-100 nm)	4.97	a	4.96	a	4.96	a	4.97	a
T ₁₂	Nano Fe @ 0.5 mg kg ⁻¹ (< 50 nm)	4.92	a	4.90	a	4.91	a	4.90	a
T ₁₃	Nano Fe @ 0.5 mg kg ⁻¹ (50-100 nm)	4.91	a	4.89	a	4.90	a	4.89	a
T ₁₄	Nano Fe @ 0.5 mg kg ⁻¹ mixed with FYM (1: 100) (< 50 nm)	4.94	a	4.94	a	4.94	a	4.95	a
T ₁₅	Nano Fe @ 0.5 mg kg ⁻¹ mixed with FYM (1: 100) (50-100 nm)	4.93	a	4.93	a	4.93	a	4.94	a
T ₁₆	Nano Fe @ 0.25 mg kg ⁻¹ (< 50 nm)	4.90	a	4.88	a	4.89	a	4.88	a
T ₁₇	Nano Fe @ 0.25 mg kg ⁻¹ (50-100 nm)	4.89	a	4.87	a	4.88	a	4.87	a
T ₁₈	Nano Fe @ 0.25 mg kg ⁻¹ mixed with FYM (1: 100) (< 50 nm)	4.92	a	4.92	a	4.93	a	4.92	a
T ₁₉	Nano Fe @ 0.25 mg kg ⁻¹ mixed with FYM (1: 100) (50-100 nm)	4.91	a	4.91	a	4.91	a	4.92	a
S.Em. ±		0.12		0.12		0.13		0.14	

Note:

FeSO₄.7H₂O @ 25 kg ha⁻¹ (12.5 mg FeSO₄.7H₂O kg⁻¹ soil) is equivalent to 5 kg Fe ha⁻¹ (2.5 mg Fe kg⁻¹)

DAI –Days after incubation

Conclusion

The incubation studies revealed that, DTPA iron in soil decreased with advancement in period of incubation. The water soluble iron increased in all the treatments up to 45 DAI and thereafter, decreased. Highest DTPA iron and water soluble iron was recorded in the treatment receiving nano Fe @ 2.5 mg kg⁻¹ mixed with FYM. The treatments which received iron oxide formulations chelated with FYM recorded higher DTPA extractable iron than their respective unchelated ones. Between the sizes, the nanoparticles having the size < 50 nm were found better than the size range of particles between 50 - 100 nm in maintaining soil DTPA iron and

water soluble iron status at all concentration ranges throughout the incubation period.

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