RESEARCH ARTICLE OPEN ACCESS

MoO_3 Nanoparticles: Synthesis, Characterization and Its Hindering Effect on Germination of Vigna Unguiculata Seeds.

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ABSTRACT
Molybdenum trioxide nanoparticles have been synthesized by sol-gel method. The X-ray diffraction analysis was done to confirm that the obtained product was MoO_3. The scanning electron microscopy was done to study the shape, size distribution and surface morphology of nanoparticles; they had a hexagonal shape with smooth surface and uniform size distribution. The functional groups were studied using Fourier transform infrared spectroscopy. The effect of MoO_3 nanoparticles on seed germination of vigna unguiculata was studied for 6 days from the day of sowing, by comparing the time taken for seeds to germinate and length of shoot with respect to time of the seeds sowed in heavy black soil whose nutrient composition was known with seeds sowed in the same heavy black soil but which was made rich with MoO_3 nanoparticles. It was observed that the MoO_3 nanoparticles hampered the germination of vigna unguiculata seeds and this restraint continued in the shoot growth also.

Keywords **–** Heavy black soil, MoO_3 nanoparticles, Seed germination, Shoot growth, Vigna unguiculata

I. INTRODUCTION

The major part of human history revolves around agriculture and its development. The integration of agriculture and science has led to great advancement in the production of food. The agro-chemical industry is a huge area and has lot of opportunities to expand vastly. Though the involvement of science in agriculture has many advantages, it also has its own disadvantages like entry of chemicals and toxic substances into the food chain, pollution of lakes, ponds and other water bodies, etc.

Nanotechnology being the new phase of science and engineering has a wide range of application even in agricultural field. The nanoparticles can be given as additional supplements to plants which can make the plants bacteria, fungi and drought resistant. As mentioned earlier integration of agriculture with science not only has pros but also cons. Similarly the involvement of nanotechnology in agriculture also has its own defects. The negative effects of nanotechnology's involvement in agriculture could be slower germination of seeds, slower growth of plant, lesser output or easier entry of chemicals into the food chain due to their extremely small size.

II. MATERIALS AND METHODS

2.1 Synthesis: MoO_3 nanoparticles were synthesized by sol-gel method. Ammonium heptamolybdate, citric acid and ammonium hydroxide were used as precursors. 1M ammonium heptamolybdate was dissolved in 100ml of double distilled water. 0.20M citric acid crystals were dissolved in 100ml of double distilled water. To the ammonium heptamolybdate solution, the citric acid solution was added slowly drop by drop under continuous magnetic stirring. Whilst stirring, ammonium hydroxide was added to the solution till the solution reached a pH of 7. The solution was then heated in a hot air oven till the supernant liquid got evaporated. The final product obtained was dark brown in color. The obtained product was finely crushed and calcinated at 700^oC for 2hrs in a muffle furnace. During calcination the product turned from dark brown to bluish grey in color.

2.2 Soil treatment: To study the effect of MoO_3 nanoparticles on vigna unguiculata seeds, heavy black soil was chosen. The concentrations of nitrogen, phosphorus and potassium in the soil were evaluated and found to be 86 Kg/acre, 27 Kg/acre and 339 Kg/acre respectively. The pH of the soil was tested and was found to be 7.9, which is slightly basic in nature. The electric conductivity of the soil was tested and found to be 0.59 milliohms/cm at 25^oC. The density of the soil was 2650 kg/m^3. Two pots were filled with 12 kg of heavy black soil each. Soil from one pot was made rich with MoO_3 nanoparticles by rigorously mixing MoO_3 nanoparticles to the soil. The concentration of MoO_3 nanoparticles in the soil was maintained at 170ppm.

2.3 Seed germination scrutiny: The effect of MoO_3 nanoparticles on germination of vigna unguiculata was studied by comparative method. Two pots were filled with 12 kg heavy black soil of each. Soil from

one pot was treated with MoO_3 nanoparticles, a concentration of 170ppm was maintained.
The scrutiny was done for six days by sowing five vigna unguiculata seeds in each pot and observing the time taken for their germination and also the length of their shoot with respect to time. During the observation natural, favorable and similar environmental conditions were maintained for either of pots. The pots were watered with 250ml of tap water each, twice a day. The average temperature during the day was 30°C and during night was 24°C, all along the scrutiny.

III. CHARACTERIZATION

3.1 X-ray diffraction: The XRD peaks were observed at 12.8°, 23.5°, 25.7°, 27.3°, 29.8°, 33.6°, 39.1°, 49.3°, 58.9° and 64.5°, when the obtained product was subjected to XRD analysis using Bruker D-scan X-ray crystallography equipment. A Cu-k-alpha radiation (1.54 A°) was used, operated at 40Kv and 30mA, with 2 ranging from 10°-80°. When the peak values were compared with JCPDS software they matched with JCPDS card no.35-0609. The comparison confirmed that the obtained product was MoO_3 and that it had an orthorhombic structure.

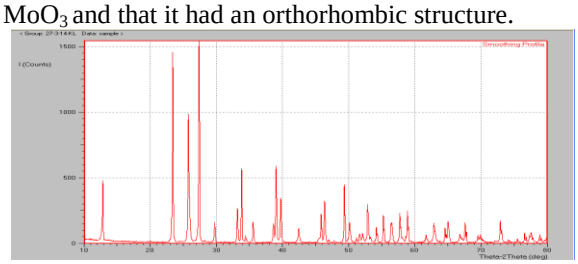

Fig. 3.1 XRD plot of MoO_3 nanoparticles

3.2 Scanning Electron Microscopy: The FE-SEM analysis was done to study the surface morphology of MoO_3 nanoparticles. The analysis was carried out at 10µm, 5 µm, 3 µm and 1 µm scale. It was clearly visible from the FE-SEM micrographs that the size distribution of MoO_3 nanoparticles was fairly uniform. The image mostly comprised of the particles that had a hexagonal structure. Very few particles with an intermittent shape have been indentified which were sparse in number. The surface of the particles with hexagonal shape was smooth where as the unevenly shaped particles had a spiked or rugged surface. Since the population of irregularly shaped particles was very skimpy compared to the particles with regular shape, it can be concluded that the MoO_3 nanoparticles had a pretty uniform shape.

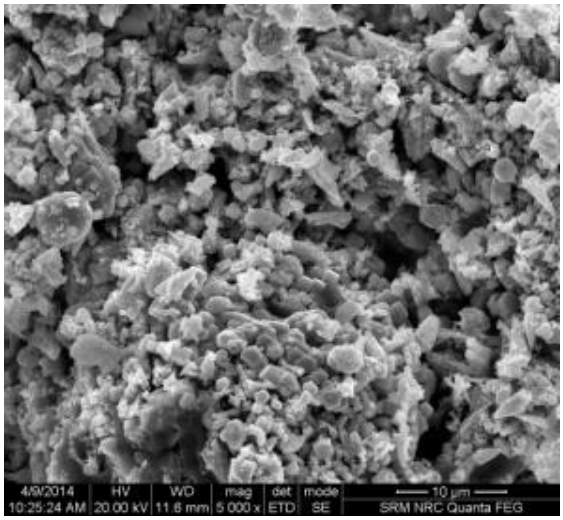

Fig. 3.2 SEM micrograph of MoO_3 nanoparticles at 10µm scale

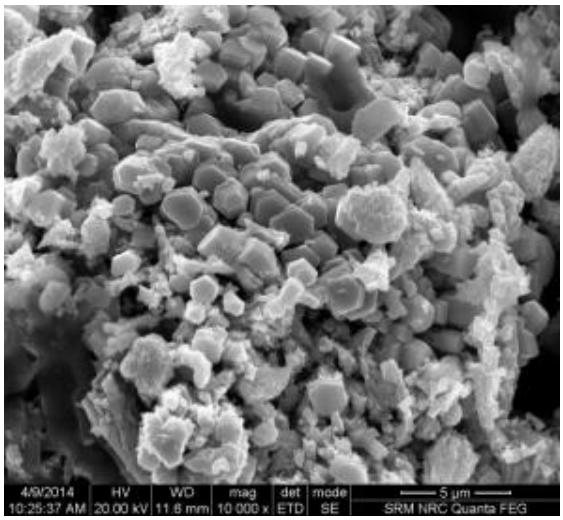

Fig. 3.3 SEM micrograph of MoO_3 nanoparticles at 5µm scale

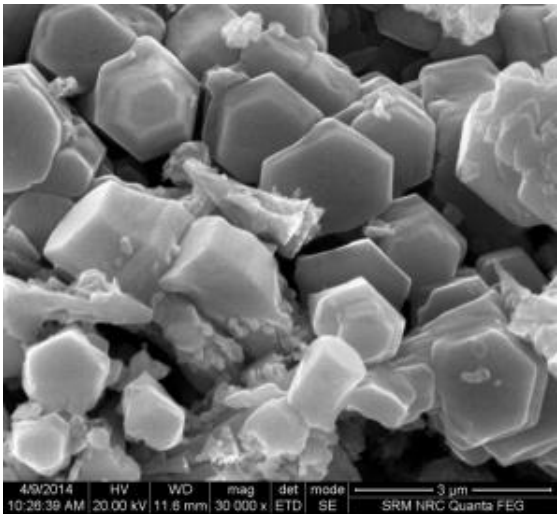

Fig. 3.4 SEM micrograph of MoO_3 nanoparticles at 3µm scale

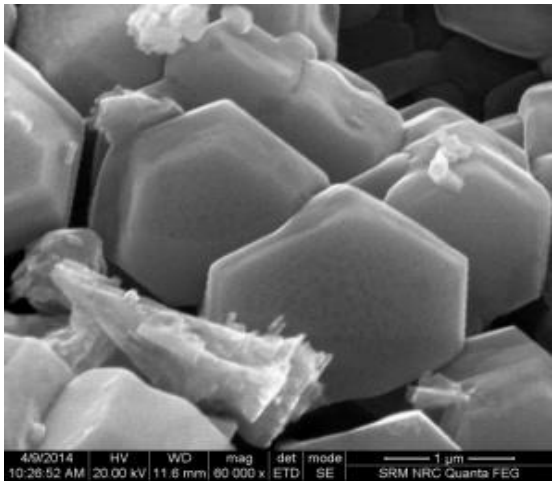

Fig. 3.5 SEM micrograph of MoO_3 nanoparticles at 1µm scale

3.3 Fourier Transform Infrared Spectroscopy: The FTIR spectroscopy was done to study the functional groups present in the obtained product. In the FTIR spectroscopy all the inorganic compound stretching peaks can be seen between 400 cm^{-1} to 1000 cm^{-1}. In the FTIR plot, a broad peak was observed at 400cm^{-1}, this peak can be corresponding to Mo-O stretching. The peaks that correspond to organic compound stretching would be seen beyond 1000 cm^{-1} till 4000 cm^{-1}. These peaks were observed at 1350 cm^{-1}, 1508 cm^{-1}, 1680 cm^{-1}, 2359 cm^{-1}, 3335 cm^{-1} and 3859 cm^{-1}, which correspond to functional group (functional group name) –C-H (alkane), C-C (aromatic), C=O (carboxylic acid), O-H (carboxylic acid), N-H (amine) and O-H (alcohol) respectively. All the above stretching are a result of using ammonium heptamolybdate, citric acid and ammonium hydroxide as precursors.

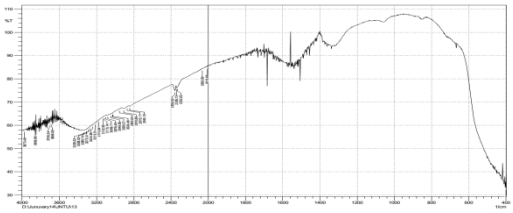
Fig. 3.6 FTIR plot of MoO_3 nanoparticles

3.4 Seed germination and shoot growth analysis: Five vigna unguiculata seeds were sown in each pot on day 0. The pots were watered and were kept under continuous observation. On day 1 and day 2 no sign of germination was seen.

On day 3 germination of vigna unguiculata seeds could be seen in either of the pots. In the pot with control soil 100% germination was seen whereas in the pot with MoO_3 nanoparticles treated soil only 60% germination was seen. The average length of the shoot was 4cm and 2.5cm for plant in control soil and treated soil respectively. There was no sight of leaves in either of the pots.

Fig. 3.7 Observation of seed germination on Day 3

On day 4, significant growth in the plant could be observed. The germination in the treated pot was unchanged at 60%. The average length of shoot was 8.9cm and 7.5cm in control soil and treated soil respectively. Leaves were first observed on day 4. Two leaves per plant in either of the pots were observed.


Fig 3.8 Observation of plant growth on Day 4

On day 5, 80% germination was seen in the pot with treated soil. The average length of the shoot was 10cm and 9cm in control soil and treated soil respectively. The number of leaves remained persistent at two per plant in either of the pots.

Fig. 3.9 Observation of plant growth on Day 5

On day 6, there was 100% germination in both the pots. The average length of the shoot was 13.3cm and 11.2cm in control soil and treated soil respectively. There were two leaves on all the plants in the pots.

Fig 3.10 Observation of plant growth on Day 6

Table 3.1 Seed germination and growth of vigna unguiculata plant in heavy black soil with respect to time

Criteria\Time	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6
Germination (%)	0	0	100%	100%	100%	100%
Average length of shoot	-	-	4cm	8.9cm	10cm	13.3cm
Number of leaves per plant	-	-	2	2	2	2

Table 3.2 Seed germination and growth of vigna unguiculata plant in soil treated with MoO_3 nanoparticles with respect to time

Criteria\Time	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6
Germination (%)	0	0	60%	60%	80%	100%
Average length of shoot	-	-	2.5cm	7.5cm	9cm	11.2cm
Number of leaves per plant	-	-	2	2	2	2

IV. CONCLUSION

MoO_3 nanoparticles were successfully synthesized by sol-gel method. The synthesized product was confirmed to be MoO_3 by studying its XRD graph. The shape, size distribution and surface morphology of the MoO_3 nanoparticles were studied by analyzing the FE-SEM micrographs. The functional groups of the final product were studied using FTIR spectroscopy.

The effect of MoO_3 nanoparticles on seed germination of vigna unguiculata was studied by sowing its seeds in two pots, one containing plain heavy black soil and the other with the same heavy black soil made rich with MoO_3 nanoparticles. The concentration of MoO_3 nanoparticles in the soil was 170ppm. The time taken for germination and average length of the shoots were studied with respect to time for 6 days. The time taken for germination was more for seeds sown in treated soil than the time taken for germination of seeds sown in virgin soil. The average length of shoots from the day of germination till day 6 was found to be more for the plants growing in virgin soil than the plants growing in treated soil. These observations revealed that MoO_3 nanoparticles have hindered the seed germination and growth of vigna unguiculata.

V. ACKNOWLEDGEMENT

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