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# SYNTHESIS CHARACTERIZATION AND APPLICATIONS OF SULPHUR NANOPARTICLES BY GREEN RUTE

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#### ABSTRACT

Sulphur nanoparticles had been prepared from sodium thiosulphate pentahydrate ( $Na_2S_2O_3.5H_2O$ ) in the presence & absence of aqueous aloevera extract &investigated the potential for enhancing the rootand shoot growth of *False daisy* (Maka) and *Pearl millet* (bajara) plants. The resulting Sulphur nanoparticles were examined by fourier transform infrared spectroscopy (FT-IR), X-ray Diffraction (XRD), &Scanning Electron Microscope (SEM). The average size of Sulphur nanoparticles was determined by XRD analysis and calculated using the Debye-Scherrer formula.

**KEYWORDS:** Sulphur nanoparticles, Green rute, for plants.

#### INTRODUCTION

Nanotechnology has been paid much attention since it can be applied in many different areas in different scientific fields such as green energy<sup>[1]</sup>, catalysis, agriculture, composites, antimicrobial agents, industrial applications, & medicines. Nanotechnology manipulation of matter on an atomic, molecular, & supramolecular scale. National Nanotechnology Initiative defines nanotechnology as the manipulation of matter with at least one dimension sized from 1 to 100 nm. Nanoparticles can be synthesized using many different methods(fig.-1) such as electrochemical, micro emulsion, cell membrane, sublimation, sedimentation, liquid phase chemical precipitation methods. [2,3,4] However, all the methods are expensive & hard to scale up in addition to needing a lot of surfactants, & being environmentally unfriendly. Environmental friendly synthesis or green synthesis is one of the best method for appllication of synthesis of nanoparticles. This method is chosen because it is easy, simple & convenient as well as, it requires less reaction time &also not harmful to environment.<sup>[5]</sup>

Green synthesis is a method of using plant extracts for the biosynthesis of nanoparticles. Synthesis of Sulphur nanoparticles is the top-down method. Many biomaterials including plantscan be used as bioreactors for the synthesis of nanoparticles. As bioreactors, plants are able to transforms inorganic metal ions into metal nanoparticles through the reductive capacities of the metabolites (such as terpenoids, alkaloids, polyphenols, tannins, &steroids) present in the plants. [7] Brassica juncea & Medicago sativa was shown to accumulate 50 nm of silver nanoparticles when silver nitrate was used

as a substrate<sup>[8]</sup>, *Iris pseudacorus* accumulated copper nanoparticles of 2 nm with substrates containing salts<sup>[9]</sup>, *Pleurotuscornucopiae var. citrinopileatus* accumulated silver nanoparticles with the size of 100 nm<sup>[10]</sup>, *Xanthium strumarium* or *Xanthium indicum Kone* accumulated gold nanoparticles with sizes ranging from 9.60 nm to 11.70 nm<sup>[11]</sup> & *Allium cepa* accumulated gold nanoparticles of 45.2 nm.<sup>[12]</sup>

Sulphur has a wide range of application in different industries & apicultural activities. Sulphur nanoparticles have many advantages over micro-Sulphur for their peculiar quantum size particles & high surface area. Sulphur acts as asoil conditioner in reducing the sodium content, & nitrogen fixing. For plants Sulphur is necessary in the formation of protein, amino acids, enzymes, vitamins & chlorophyll. Also Sulphur helps the plants resistance to disease. [13] In this, we examined the synthesis of Sulphur nanoparticles& its beneficial effect on growth of shoot & leaves of plants. Sulphur nanoparticles are non-metal & are also a safe element. As a safe element, Sulphur has the ability to form organosulphur compounds in cells. Aloe vera has been known to contain polysaccharides which have therapeutic properties such as anti-bacterial, anti-viral, anti-fungal, anti-oxidant.

Agriculture farming is the foundation of most developing economics; roughly 60% of the population relies upon horticulture for their work. Indian farming division represents 18% of India's total national output (GDP) & gives work to 50% of theworkfaceof the nation. Nanotechnology impacts the horticulture efficiency with the assistance of the Nano-fertilizer, Nano-pesticides or

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Nano-herbicides which act as a smart delivery system to plant, likewise the different industries making definitions with Nanoparticles (100-250 nm) to improve their activities by expanding nanoparticles solubility in water. Here we use Sulphur nanoparticles synthesized by using aqueous extract of aloe vera gel and its effect on growth of plant. The soil is very important component of the land & it is the mixture of organic matter, minerals, gases, liquids & organism that support the life on the earth.

Soil well-being has been defined as the limit of soil to work as a living framework, with the environment & land us limits, to continue plant & living system efficiency, keep up or upgrade water & air quality & advanced plant & organism's well-being. Pesticides enhance the quality of soil which helps to improve the growth of plants. fertilizer is the material of natural or synthetic origin that is applied to soil or to plant tissues to supply one or more plant nutrients essential to the growth of plants. May sources of fertilizers exist, bith natural & industrially produced. It enhances the growth of plant. This goal met in two ways, the traditional one being additives that provide nutrients. The second mode by which some fertilizers act is to enance the effectiveness of the soil by modifying its water retention & aeration.

# **EXPERIMENTAL**

Preparation of aqueous extract of Aloe vera: 25 gm of fresh odorless& clear liquid i.e. aloe vera gel was extracted from innermost part of the leaf. It was mixed with 250 ml distilled water& boiled it for about 30 minutes. The mixture of aloe vera was then cooled at room temperature. It was then filtered using Whatman Paper no. 41 for the purpose of removing raw materials from the extract. The aqueous aloe vera extract was then centrifuged for 5 minutes at 1500 rpm for the purpose of removing heavy biomaterials. The filtrate obtained by decantation was stored in a refrigerator for further purpose.

**Phytochemical screening of garlic extract:** The method described by Harbone<sup>15</sup> were used for the phytochemicals test. The extract was tested for the presence of alkaloids, tannins, saponins, cardiac glycosides, steroids, phenols & flavonoids.<sup>[14]</sup>

**Synthesis of Sulphurnanoparticles:** With slight modifications, Sulphur nanoparticles were synthesized by the method developed by Nida<sup>3</sup>. 24.8 g of sodium thiosulphate pentahydrate (Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>.5H<sub>2</sub>O) was dissolved in thepresence& absence of 250 ml aloe vera extract with mild stirring at room temperature for 10 minutes. Then the mixture was diluted with 250 ml distilled water. Immediately, added 10% hydrochloric acid (HCl) drop by drop using pipette under constant stirring for allowing the Sulphur precipitations uniformly. The suspendedsolid Sulphur particles (in the presence & absence of 250 ml aloe vera extract) obtained were centrifuged for 50-60

minutes at 3000 rpm at airtemperature. The supernant was removed & the Sulphur precipitate was washedwith distilled water repeatedly & then washed with absolute ethanol in order to remove any biological material or impurities from the product. After purification Sulphur nanoparticles were dried at room temperature & stored for the next analysis. The disproportionation reaction between sodium thiosulphate pentahydrate & hydrochloric acid is as bellow.

 $Na_2S_2O_3.5H_2O_{(s)} + 2HCl_{(aq)} \rightarrow 2NaCl_{(aq)} + S_{(s)} + SO_{2(aq)} + 6H_2O_{(l)}$ 

# Application of sulphur nanoparticles on the plants Preparation of seedbed for the seed germination

Removal of debris: The debris from soil such as fungal spores, insect eggs, stones, unwanted wasteare removed for reducing soil population.

Levelling of soil: leveling of soil was done for the top fertile soil from being away by strong winds or washed away by rain water. It helps in the uniform distribution of water in the fields during irrigation & prevents loss of moisture.

Parting the soil: The levelled soil is further divided into five different parts & labelled them to recognize the purpose of parting.

Watering the bed: waterd theprepared bed & made itmoist for further procedure.

Fertilizing the soil: fertilizer was added in the three parts of soil only for further procedure.

Adding sulphur nanoparticles: Prepared Sulphur nanoparticles with aloe vera were added in two parts of soil one in with fertilizer & other in without fertilizer parts that we had prepared. Then, Sulphur nanoparticles without aloe vera were added in two parts of soil onein with fertilizers & other in without fertilizer. One part of soil is remaind as it is with only fertilizer.

Sowing the seeds: In each part of the soil we sowed the seeds of maka (false daisy) & bajara (prearl millet).

Hence, the parts of bed were as follows

Part 1: seeds+ Sulphur n.p. without aloe vera + fertilizer

Part 2: seeds+ Sulphur n.p. without aloe vera

Part 3: seeds+ Sulphur n.p. with aloe vera + fertilizer

Part 4: seeds+ Sulphur n.p. with aloe vera

Part 5: seeds+ fertilizers

Experiment were carried out under greenhouse. The False Daisy (maka) & Pearl Millets (bajara) were used for the experiment. The response of these plants to Sulphur nanoparticles (with & without Aloe vera) were studied for 10 days.

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#### Shoot width & plant height

Shoot width was taken with the help of the Vernier caliper & plant height was measured by thread & scale for 10 days.

#### Fresh & dry weight

The weight of fresh leaves & shoot of seedlings were taken on electric balance. Later, the leaver & shoot were heated in electrical oven at 60°C for a 3 hours then weight of dry matter was recorded.

# Characterization of Sulphur nanoparticles

Fourier Transform Infrared (FT-IR) Analysis: FT-IR spectroscopy (using an) was used to determine the Fourier Transmission Infrared (FT-IR) spectra for aqueous aloe vera extract, sodium thiosulphate pentahydrate, & Sulphur nanoparticles synthesized in the presence & absence of aqueous aloe vera extract. The FT-IR spectra were recorded in the range of.

**X-ray Diffraction (X-RD) Analysis:** The average size of crystalline Sulphur nanoparticles that were synthesized in the presence & absence of aqueous aloe vera extract were determined by X-ray Diffraction, (X-RD) (using a)& calculated using the Debye-Scherrer formula.

Scanning Electron Microscopy (SEM) Analysis: The Scanning Electron Microscope (SEM) analysis of the synthesized Sulphur nanoparticles was done using a.

#### RESULTS

# Phytochemical screening of aqueous Aloe vera extract

The phytochemical screening of aqueous Aloe vera extract is as shown in Table no-1. The table shows the qualitative phytochemical analysis in which Alkaloids, tannins, sterols, flavonoids, phenol were present & saponins, cardiac glycoside were absent.

Table no-1.

Name of test	Aqueous extract of Aloe vera	
Alkaloids	+	
Saponins	-	
Tannins	+	
Sterols	+	
Cardiac glycoside	-	
Flavonoids	+	
Phenols	+	

(+):present, (-): absent

# Green synthesis of Sulphur nanoparticles

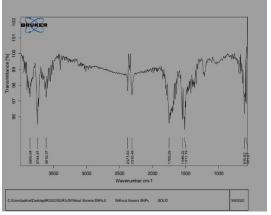
The yield of the Sulphur nanoparticles is shown in table.

Method of synthesis	Na <sub>2</sub> S <sub>2</sub> O <sub>4</sub> .5H <sub>2</sub> o (gram)	Sulphur nanoparticles (gram)	Percentage of yield (%)
In the presence of aloe vera extract	24.80	1.81	7.19
In the absence of aloe vera extract	24.80	1.98	7.95

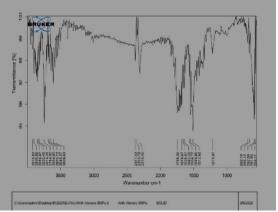
# FT-IR anlysis of aqueous aloe vera extract and Sulphur nanopaerticles

FT-IR analysis was performed in order to identify the potential of aloe vera extract as a bioreactor in the reduction of sulphur ions & the synthesis of sulphur nanoparticles. FT-IR analysis was conducted on a

control(aqueous aloe vera extract), starting matrial (sodium thiosulphate pentahydrate,  $Na_2S_2O_4.5H_2o$ ), & the Sulphur nanoparticles in the presence & absence of aloe vera extract. The FT-IR was recorded in the range of  $4000-550~\text{cm}^{-1}$ .



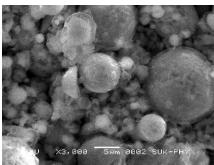
a.FT-IR spectra of sulphur nanoparticles without aloe vera.



b.FT-IR spectera of sulphur nanoparticles with aloe vera.

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The FT-IR spectrum for sulphur nanoparticles with aloe vera extract showed peaks at 3814.28; 3645.27; 2353.78; 1746.92; 1512.65; 1215.97 & 592.59cm<sup>-1</sup>.The FT-IR spectra peaks at 3814.28 cm<sup>-1</sup> are represented the O-H stretching vibration, the presence of carbohydrates & amino acids. The spectra at 1746.92cm<sup>-1</sup>& 1512.65 cm<sup>-1</sup> peaks represents amide C=O stretching mainly: proteins & spectra peaks at 1215.97 cm<sup>-1</sup> represents the C-O single bonds. However, the FT-IR spectrum of sulphur nanopar6icles showed the peaks at 3744.81; 3610.37;



a. SEM analysis of sulphur nanoparticles with aloe vera.

#### Effect of sulphur nanoparticles on growth of plant

Effect of sulphur nanoparticle on diameter of stem & height of growth of False daisy (Maka) and Pearl millet (bajara) plants. The growth of these plants to Sulphur nanoparticles (with & without Aloe vera) was studied for 10 days.

#### CONCLUSION

Sulphur nanoparticles have been successfully prepared from sodium thiosulphate penta hydrate in the presence and absence of aqueous aloe vera extract. SEM results showed that sulphur N.P synthesized in the presence of aqueous aloe vera extract had clear and smooth spherical shape. The suspended sulphur nano particles formed by this rout indicated that aloe vera extract act as capping agent and stabilised the SNPs. NP of sulphur are quickly transported through the plant and included in the metabolic process through forming organosulphur compound which are neccesary to plant growth . The plant with SNPs with aloe vera grew faster (with 15.1 cm height) and had more leaves and thicker stem (11.5cm). This conclude that the SNPs with aloe vera are useful for plant growth.

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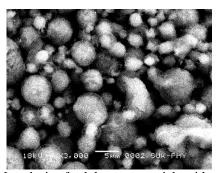
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1750.29; 1511.193814.28; 3645.27; 2353.78; 1746.92; 1512.65; 1215.97 & 592.59cm<sup>-1</sup>.

# SEM analysis of synthesized sulphur nanoparticles

In the SEM analysis, the suspended sulphur nanoparticle in sterile aqadest were examined with SEM, using a JSM-6360 machine at 18kV with a magnification of 5.000 & 50.000x.SEM.

images of sulphur nanoparticles synthesized in the presence & absence of aloe vera extract are.



b. SEM analysis of sulphur nanoparticle without aloe vera.

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