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Synthesis of silver nanoparticle by using 3-methyl pyrazol 5-one (chemical reduction method) and its characterization

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Abstract

The synthesis of metal nanoparticles is in vogue due to their applications in various fields. We synthesized nanoparticles by chemical reduction method. In this study, first we prepared reducing agent (3-methyl pyrazol-5-one) by chemical method and that reducing agent is then used for the preparation of silver nano particles. The characterization of silver nanoparticles was done by using UV-Visible spectrophotometer, FTIR, SEM and XRD. It was found that the growth of Escherchia coli and Klebsiella pneumoniae was inhibited by this synthesized silver nanoparticles. The average particle size of the silver nano particles calculated from XRD spectra is about 17.68 nm.

Keywords: Nanoparticles, UV-Visible spectrophotometer, FTIR, SEM and XRD, Antibacterial activity

Introduction

The preparation and characterization of novel nano structures such as cubes, disks, plates, prisms, wires, rods and triangle etc. The various metal are used for the synthesis of nanoparticles such as Silver, Tin, Cadmium, Zinc, Copper and Gold have come a long way of various investigation in many laboratories and there uses in photography, catalysis, biological labeling, superconductors, photonics, sensor, antimicrobial and antibacterial activities etc. Chemical reduction is the most frequently applied method for the preparation of the stable silver nanoparticles. The chemical reduction methods are probably the most versatile, economical and easy to control the shape and size of nanoparticles. The 3-methyl pyrazol-5-one is a one of the heterocyclic compound and this compound shows therapeutic importance. Pyrazolone derivatives have been used as analgesics, antimicrobial agents, fungicides and hypoglycemic agents. Some of them have been tested as potential cardiovascular drugs. They inhibit the production of TNF- α and decreases level of pro-inflammatory cytokines and thereby reduce inflammation and prevent further tissue destruction diseases such as Rheumatoid Arthritis, Osteoarthritis and Crohns disease. It is believed that the redox chemistry of 3-methyl pyrazol-5-one plays an important role in the pharmacy and the silver nanoparticles may be useful. In this work we focus, for the first synthesis of 3-methyl pyrazol-5-one, synthesis of silver nanoparticles and the reduction of Ag^+ ions by 3-methyl pyrazol-5-one.

2. Experimental:

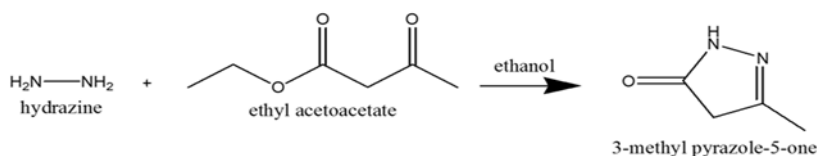
All chemicals used in the present study are of high purity.

2.1. Synthesis of 3-methyl pyrazol-5-one

5.2 ml (0.04 mole) of ethyl acetoacetate was taken in 25 ml beaker and stirred magnetically during slow drop wise addition of a solution of 2 ml (0.04 mole) of $NH_2NH_2 \cdot H_2O$ (Hydrazine hydrate) in 3.5 ml ethanol. The temperature of the reaction mixture increased during reaction so that temperature was regulated at $55^\circ C$. The reaction mixture was cooled in an ice bath to complete the crystallization. After standing for some time for complete of crystallization, it was filtered and the solid was washed with cold alcohol, dried and used for next step.

(Yield = 76.21% & melting point = $223^\circ C$)

Reaction



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2.2. Synthesis of silver nanoparticles

Boil 0.5 gm of 3-methyl pyrazol-5-one in 75 ml distilled water. It is completely soluble and filters it with Whatmann No: 41 filter paper and then, 10 ml of the aqueous solution of 3-methyl pyrazol-5-one was added 90 ml of 0.1 M AgNO₃ solution. The solution turned to faint violet colour indicating the formation of silver nanoparticles. Then solution incubated for 24 hrs, the resulting solution was centrifuged at 10000 rpm / 7 min, the mixture was collected after discarding the supernatant. The collected silver nanoparticles were allowed to dry in a watch glass.



Fig 1.a) solution of reducing agent and AgNO₃ b) after addition of metal solution and reducing agent

2.3 Characterization of synthesized nanoparticles

The structural and chemical composition of silver NPs were analyzed by SEM (JEOL JSM-6360) and XRD (Bruker D8-Advanced Diffractometer) and the reduction of Ag⁺ ion in aqueous solution were monitored with the help of UV-Visible spectroscopic analysis method. UV-Visible spectroscopic analysis of silver nanoparticles was carried out as a function of time needed for reduction at room temperature on model UV-2100 series and FTIR (JASCO 4100) was used to study the optical properties of silver NPs.

3. Result and Discussion

3.1. Characterization of silver nanoparticles

The characterization of silver Nano particle's in examine XRD pattern (Fig.2.) of silver nanoparticles, the prominent peaks at $2\theta=32.04$, 37.99 , 44.08 represent the (320), (111) and (211) Bragg reflections of the Face Centered Cubic structure of silver nanoparticles respectively. The size of silver nanoparticles was also determined from X-ray line broadening

using the Debye-Scherrer formula given as $D=0.9\lambda/\beta \cos\theta$, where D is the average crystalline size (Å), λ is the X-ray wavelength used (nm), β is the angular line width at half maximum intensity(radians) and θ is the Bragg angle(degrees). For (111) reflection at $2\theta=37.99$ for $\beta=0.475$ radians, $\lambda=1.54\text{Å}$ and $\theta=18.99$. The average size of silver nanoparticles is estimated to be around 17.68nm.

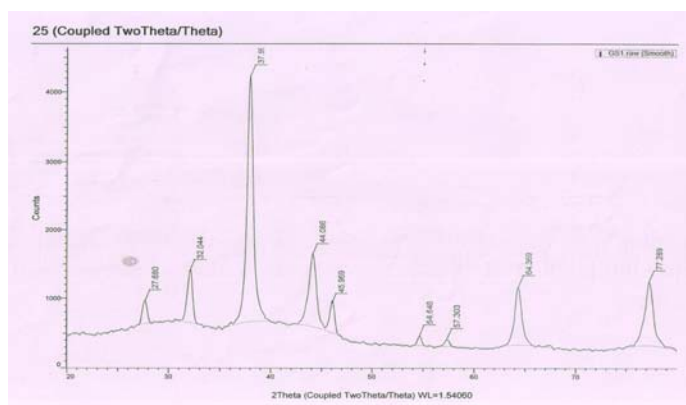


Fig: 2 XRD analysis of silver nano particles

SEM images (Fig.3) of the sample obtained from the silver solution prepared at room temperature, confirms the existence of very small and uniformly wire nano particles size. This could have contributed for the variation in particle size.

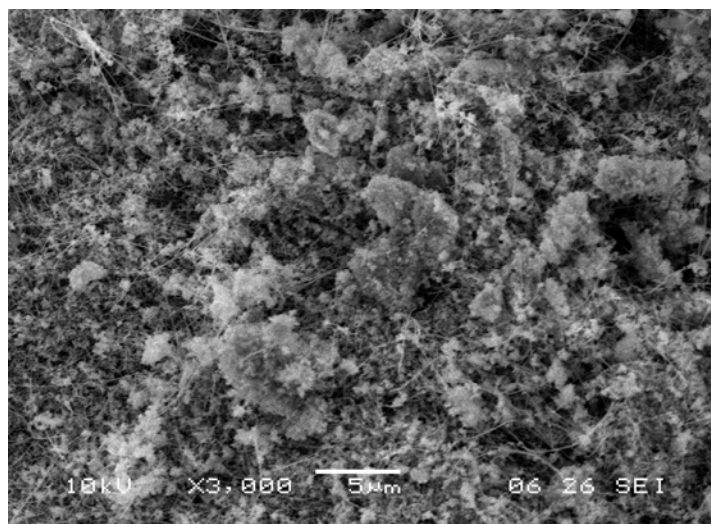


Fig: 3 SEM analysis of silver nanoparticles

The colorless silver nitrate solution forms the violet color (Fig.1.b) indicates the formation of silver nanoparticles. The appearance of the violet color was due to the excitation of the surface Plasmon vibrations, which are in the range of 400-500 nm (Sastry et al., 1997). The UV-Visible spectra (Fig.4) of nanoparticles having Plasmon resonance at 457 nm. From FTIR analysis (Fig.5) the peak around 2358.52 cm^{-1} , 1855.19 cm^{-1} , 1501.31 cm^{-1} and 1651 cm^{-1} corresponds to N-H bond vibration and C=O stretching frequency in amide group respectively.

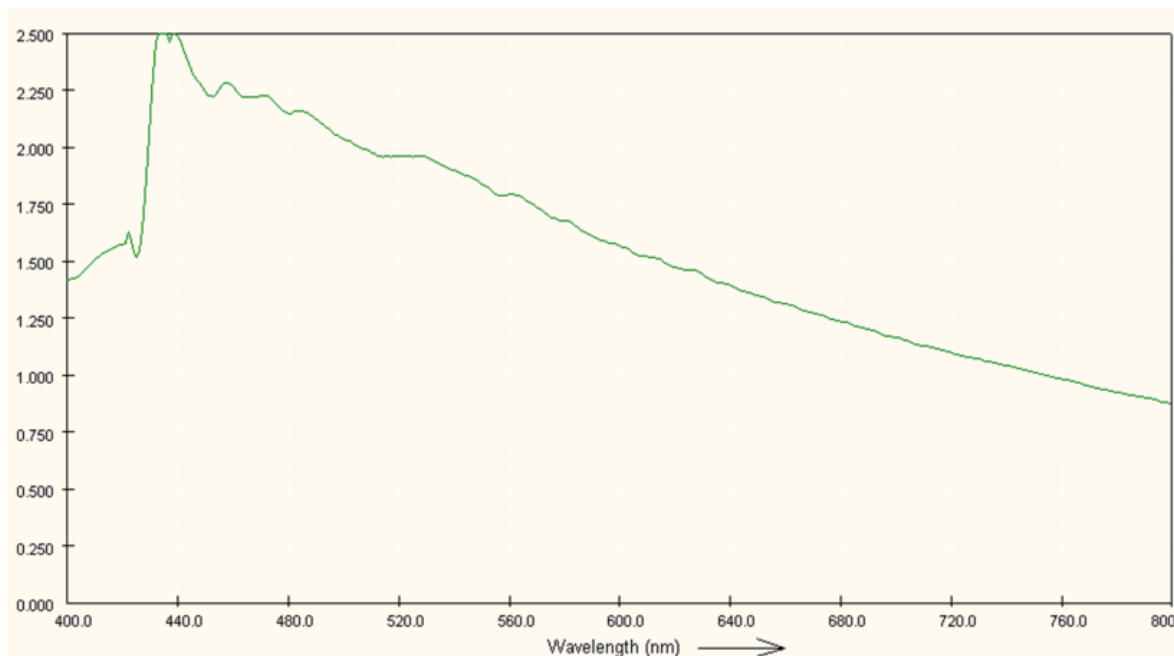


Fig: 5 UV Visible Spectra of Silver nanoparticles

The silver nanoparticle synthesized by 3-methyl pyrazol-5-one showed growth inhibitor effects against *E. Coli* and *K.pneumoniae* using disk diffusion method. The agar Petri dishes for antibacterial activity were prepared by Boiling 2.28gm of nutrient agar in 100ml water till it melted down completely. The agar solution was inoculated for 30 minutes. After inoculation, the bacterial strain(*E. coli* and *K. pneumoniae*) were poured with different concentration of AgNPs (5,4,3,2 µg/ml) in agar plates and spread evenly. The prepared agar plates with Bacterial strain and AgNPs were then kept at 37°C for 24 hours. The antibacterial activity of AgNP from 3-methyl pyrazole-5-one was checked against *E. coli* and *K. pneumoniae*.

The synthesized silver nanoparticle were Zone of inhibition was found to be in the range of 07-10mm for *E. coli* and 8-10mm for *K. pneumoniae* shown in fig5. The exact inhibition of bacterial growth reported in this study is independent on the concentration and number of nanoparticle in medium.

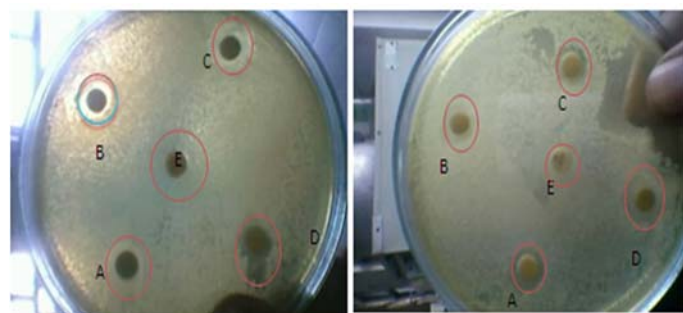


Fig5-Bacterial growth inhibition against bacterial strains a) *E.Coli*, b) *K.Pneumonia* A)5 B)4 C)3 D)2 µg/ml AgNP E) Amoxicillin

Table: 1 zone of inhibition (mm) of chemically synthesized AgNPs against bacterial pathogens

Conc. Of AgNPs (µg/ml)	<i>E.Coli</i> (zone of inhibition in mm)	<i>K.Pneumoniae</i> (zone of inhibition in mm)
5	08	08
4	10	09
3	07	09
2	07	10
Amoxillin(standard)	07	08

4. Conclusion

We found the new reducing agent which is easily used for the synthesis of nanoparticles having the average particle size 17.68 nm. The resulting nanoparticles solution displays the characteristic Plasmon absorption band for nanoparticles at 457 nm. Our results suggest that 3-methyl pyrazol-5-one is one of the best reducing agent. The synthesized silver nanoparticles were Zone of inhibition was found to be in the range of 07-10mm for *E. coli* and 8-10mm for *K. pneumoniae*. We believe that the silver nanoparticle has great potential for application in catalysis, biomedical and pharmaceutical industries.

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