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THE PHYTO CHEMICAL ANALYSIS OF GREEN SYNTHESIS AND CHARACTERISATION OF OPUNTIA DILLENII WITH FRUIT DECORATED BY SILVER NANO PARTICLES.

Prof. M.E.Rani*

PROFESSOR, HEAD & BOS, CHAIRMAN.
DEPARTMENT OF CHEMISTRY.
RAYALASEEMA UNIVERSITY, A.P, 518007 INDIA.

ABSTRACT:

Opuntia dillenii is an important medicinal shrub growing under desert and dry conditions. Though almost all of its parts are used in traditional systems of medicines, stem, fruit, flower, roots are the most important parts, which are used medicinally. Specially the fruit is considered a refrigerant, and is said to be useful in gonorrhoes. In the Deccan the baked fruit is given in whooping cough. A syrup of the fruit appears to increase the secretion of bile when given in teaspoon full doses three or four times a day and to control spasmodic cough and expectoration. This article gives an account of updated information on its Phyto chemical analysis revealed the presence of alkaloids, phenols, Flavonoids, terpenoids, steroids and glycosides. In this present study, we have synthesized Silver Nano particles using ***Opuntia dillenii*** is used as capping and reducing agent in the synthesis of Silver Nano particles. The formation of Nano particles was confirmed by UV-Visible, FT-IR, XRD and Scanning electron microscopy (SEM).This green method is simple, rapid, Echo-friendly, and reliable and it may have a potential use in the biomedical applications. In the future, selection of such plants may create a new plat-form for realizing the potential of herbal medicines in Nano-science for drug delivery.

KEY WORDS

Opuntia dillenii, Fruit, phytochemical screening fruit extract, Green Synthesis of Silver Nano Particles, Fruit Extract, UV -Visible, FT-IR, XRD, SEM.

ABBREVIATIONS

UV-Visible –Ultraviolet Visible spectroscopy

FT-IR-Fourier Transforms Infra Red Spectroscopy

XRD –X-ray diffraction

SEM.-Scanning electron microscopy

INTRODUTION:

Medicinal plants contain some organic compounds which provide definite physiological action on the human body. Secondary metabolites are chemically and taxonomically extremely diverse compounds with obscure function. They are widely used in the human therapy secondary metabolites are the substance produced by plants as defence chemicals. They include alkaloids, flavanoids, essential, oils, phenols, saponins etc. Recently, many pharmaceutical companies have spent a lot of time and money in developing natural products extracted from plants, but the information availabie is quite meagre. The present study was carried out to identify the active chemical priniciple comparision of Opuntia dillenii

Opentia dillenii (Ker-Gaw) Haw family cactaceae commonly known as pear bush, prickly pear, mal rachette or tuna,is a succulent shrub growing under desert and dry conditions.It is native to the American continent and the West Indies, but recently due to cultivation it has become widely distributed throughout Canary Islands, Southern and Eastern Africa ,Pakistan, India and Australia Opuntia dillenii is a rich source of dietary fibres, natural colorants and antioxidant vitamins and therefore, used as a food because of their edible fruit pharm acological evaluation Opuntia has shown its efficacy as antihyperlipidemic, antiviral anti-inflammatory antidiabetic antiulcerogenic activity. It has also been reported to protect nerve cells and used for the treatment of Alzheimer's disease, Parkinson's disease and stroke. In recent years, there has been a global trend toward the use of natural resources as antioxidants and functional foods.Two characteristic historical examples are the *Opuntia dillenii* plantations of srirangapatam(India).in the first case,the Ruler of Mysore,Tippu Sultan(1750-17990),reinforced the fortification around his residence with the cactus because of its form idable spines.Secondly ,in 1930 the Imam established the cactus near his castle in order to use the purple coloured juice as ink. *Opuntia dillenii* a wild xerophyte is abundant in Himalyas, believed to be of American origin and a native of India. Traditionally ,the plant is used in the treatment inflammation, hypoglycaemic, stomachuiceration, Neuro-protection through antioxida action,viradisease, disease, burms, bronchial, asthma and digestive problems throughout the world.Phytochemistry study was undertaken in fruits and revealed the presence of Phenols, Alkaloids, Flavonoids Terpenoids, Steroids,Glycoredes.

MATERIALS AND METHODS:

COLLECTION OF PLANTS:

THE Opuntia dillenii fruit collected in the Rayalaseema University Campas. PREPARATION OF PLANT EXTRACT:Peroclation procesec the opuntia dillenii fruit were ground using mixer grinder for the percolation process, the mearted plant ground were soaked in solvent such as Methanol, Ethyi acetate,Aqueous individually.

Extraction was done by soaking one part of plant ground to three parts of liquid solvent(1:3) and kept for pereolation process for 3-5 days then the crude extracts were filtered using what man NO.1 filter paper, evaporated and concentrated under room temperature and used the deposited cruke meterail for phytochemical analysis.

METERIALS AND METHODS

1) Collection and Identification of Plant Meterials :

The plant material opuntia dillenii were collecte form Rayalaseema Univerisity in Kurnool, A.P. This opuntia dillenii identified by the botony departm.

1) Preparation of Plant extract: Percolation Process :

The dried opuntia dillenii were powdered using mixer grinder. For the percolation process, the macerated fruit powders were soaked in solvents such as Mthanol, Ethyi acetate , Aqueous individually.Extraction was done by soaking one part of fruit powder to three parts of liquid

solvent(1:3) and kept for percolation process for 3-5 days. Then the crude extracts were filtered using whatman No.1 filter paper, evaporated and concentrated under room temperature and used the deposited crude materials for phytochemical analysis.

PHYTOCHEMICAL SCREENING

Phytochemical analysis of solvent extracts of the opuntia odillenii fruit samples was carried out using standard qualitative methods following the methodology of Harborne(1973) Trease and Evans(1989) and Sofowara(1993).

Tests For Alkaloids

a) Mayer's test:(Potassium Mercuric Iodide Solution:

The extracts were treated with Mayer,s reagent . The formation of a yellow cream precipitate indicates the presence of alkaloids.

b) Wagner's test:(Solution of iodine in Potassium iodide).

Few drops of Wagner's reagent were added by the side of the test tube to 1 ml of extract. A reddish-brown precipitate is produced.

c) Hager's test:(Saturated solution of Picric acid)

To the extract solution, add few dropes of Hager's reagent, Yellow precipitate is produced.

Test for Phenolic Compounds:

Ellagic Acid Test:

The test solution was treated with a few drops of 5% glacial acetic acid $NaNO_2$ solution.The solution tumeccurred in muddy or Ngier the extract.It indicates the of Phenol solution.

Ferric Chloride Test :

To 1 ml of solvent extracts, 3 ml of distilled H_2O was added. To this, a few drops of neutral 5% $FeCl_3$ solution was added. Formation of a dark green colour indicated the presence of Phenolics.

Hot Water test:

Deep the mature fruit part in a beaker containing hot water warm it for a minute of black or brown colour rinr at the junction of Deeping indicates the presence of phenols.

Lead acetate test :

3 ml of 10% lead acetate solution was added to 1 ml of the extract. Appearance of bilky white precipitate confirms the presence of phenolic compounds.

Test For Flavonoids:

A.Shinda's test;In a test tube containing 0.5ml of the extract 10 drops of con Hcl followed by a small piece of magnesium were added. Formatinon of pink,reddish or brown colour indicated the presence of Flavonoids.

B.Ferric chloride test;

Test solution with a few drops of ferric chioride solution show intense gree colour.

C.Zinc-Hydrochloric acid reduction test;

Test solution with zinc dust and a few drops of con.hydrochloric acid shows magenta red colour.

D.Alkaline reagent test;

Test solution when treted with sodium hydroxide solution,shows an in the intensity of Yellow colour which becomes colouless on the addition of a few drops of dilute acid.

E.Lead acetate solution test;

Test solution with a few drops of lead acetate (10%) solution gives a yellow precipitate.

Test for Tannins:

To 1 ml of the solvent extract, few drops of 1% $FeCl_3$ solution were added. The appearance of a blue, black, green or blue green precipitate indicated the presence of tannins.

TEST FOR TERPENOIDS

Liebermann-Burchard's test(LB test);

Fruit extract was dissolved in acetic anhydride,heated to boiling,cooled and then 1ml of con sulphuric acid was added along the side of the test tube.Formation of a violet colored ring indicated the presence of triterpenoids.

Salkowski Test: To 1 ml of the solvent extract, 2 ml of chloroform was added. Then 3 ml of conc.H_2SO_4 was added carefully to form a layer. A reddish brown coloration of the interfaces indicates the presence of terpenoids.

Test for Cardiac glycosides

Keller-Kilani Test:

The extract was dissolved in glacial acetic acid containing traces of $FeCl_3$. The tube was then held at an angle of 45° and 1 ml of Conc.H_2SO_4 was added along the sides of the tube. Formation of a purple ring at the interface indicates the presence of cardiac glycosides.

Test for Steroids:

A.Liebermann-Burchard' test;

2 ml of acetic anhydride was added to 0.5 g ethanolic extract of each sample with 2 ml H_2SO_4. Change in colour from violet to blue or green indicates the presence of steroids.

B.Salkowaski test;

2ml of extract was shaken with chloroform,to the chloroform layer sulphuric acid was added slowly by the side of test tube. Formation of red colour indicated the presence of steroids.

Phyto chemical result obtained from opuntiadillenii fruit

Phytochemical constituents	Mthanol extract	Ethyl acetate extract	Aqueous extract
Alkaloids			
Mayer's test	++	++	++
Wagner's Test	++	++	++
Hager's Test	+	+	+
PHENOLS			
Ferric chloride test	_	_	_
Ellagic Acid test	++	++	++
Lead acetate test	+	+	+
Hot water test	_	_	_
FLAVONOIDS			
Shinoda test	++	++	--
Ferric chloride test	-	_	-
Zinc-HCireductiontest	++	++	_
Alkalinereagent(NaOH)	+	-	++
Lead acetate solution test	++	++	_
Ammonia test	+	-	+
Test for steroids	+	-	+
Test for Tannins	_	_	_

Test for Terpenoids			
Liebermann-Burchard's test	++	++	_
Salkowaski reaction	++	++	_
Keller-Killiani test	++	++	_

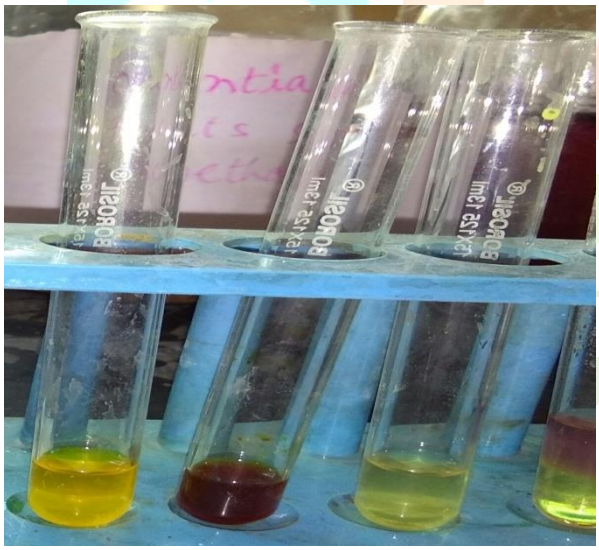


Fig:Aqueous extracts

Fig:Methaonal extract

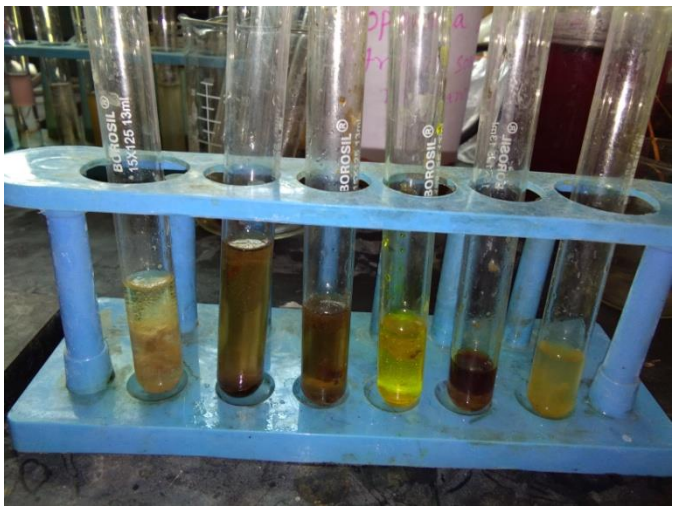


Fig:Ethyi acetate extract

CHARACTERIZATION BY USING OPUNTIA DILLENII FRUIT EXTRACT

METERIALS AND METHODS

1.Preparation of plant extract:

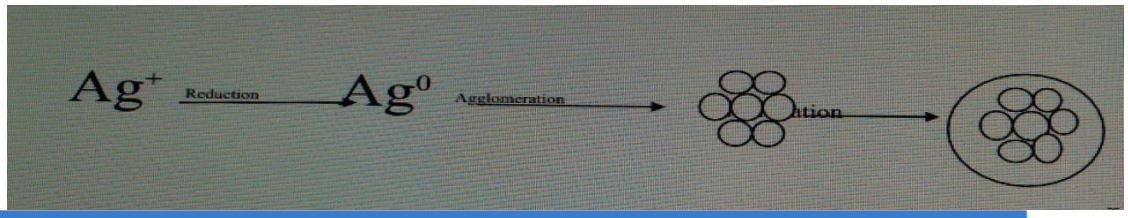
Taking 50 g of went opuntia dillenii fruit ground using mixer grinder. The macerated plant extract was done by soaking with 150 ml double distilled water, one part of plant powder to three parts of solvent water(1:3) and then boiling the mixture and refluxed for 1h at 80^{o}c and cooled that mixture. This cooled mixture was filter by Buchner flask 250 ml with Buchner funnel. Then collected filtrate is dark purple in colour. The filtrate was stored in -4^{o}c in the refrigerator, for further bio synthesis process.

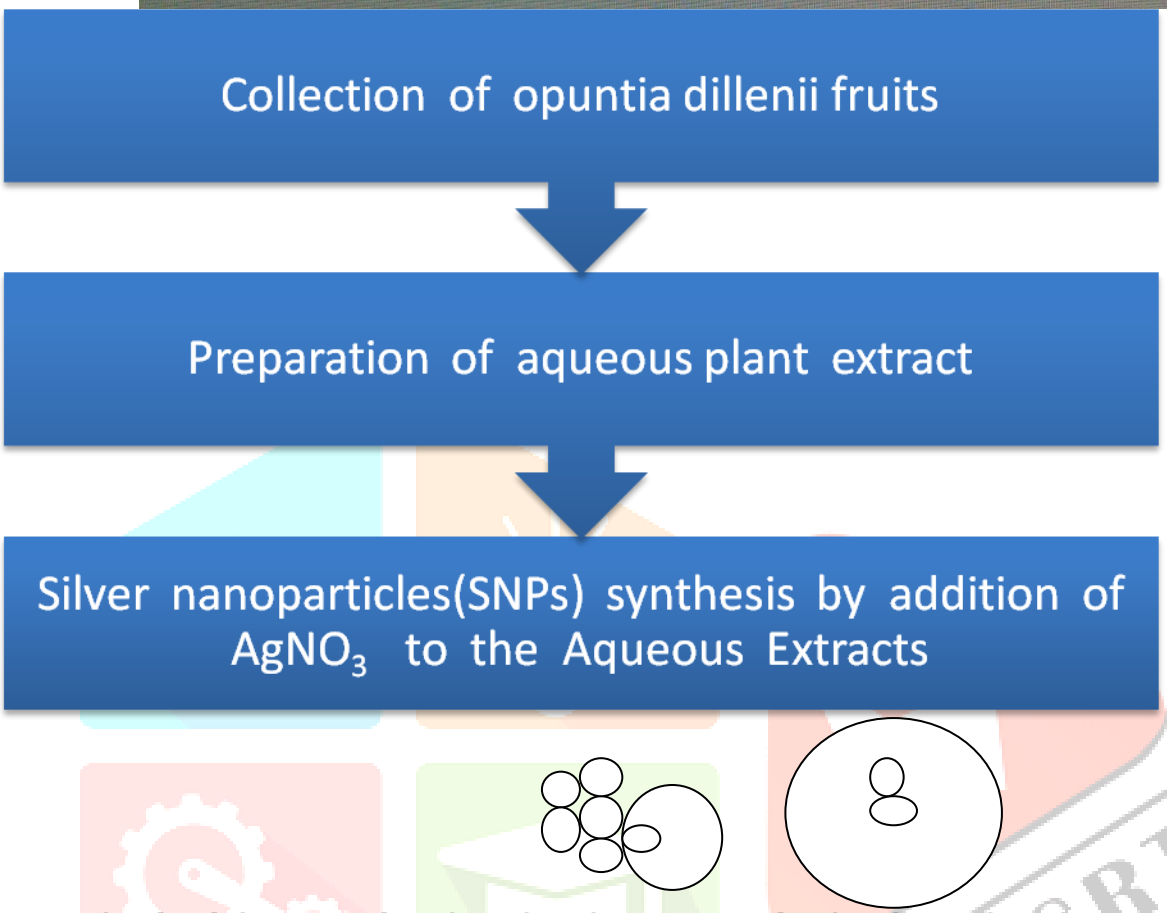
2.CHEMICALS:

Silver Nitrate($AgNO_3$) was purchased from Sigma Aldrich. Bangalore. India. Double distilled water was used throughout the experiment. All other chemicals were of analytical grade.

BIO SYNTHESIS OF SILVER NANO PARTICLES

Bio synthesis of silver nanoparticles includes, 50 ml supernatant of opuntia dillenii fruit extract was added 100 ml of 1mM of aqueous solution of $AgNO_3$(1mM) at room temperature and stirred for 1h. The reaction mixture flask was kept in dark. Finally, the colour of solution changed from dark purple colour to drack brown colour. That confirm the formation of silver nano particles. Further the colloidal solution was Ultra centrifuged at 8000 rpm for 20 min and the supernatant and solid was collected for further studies.

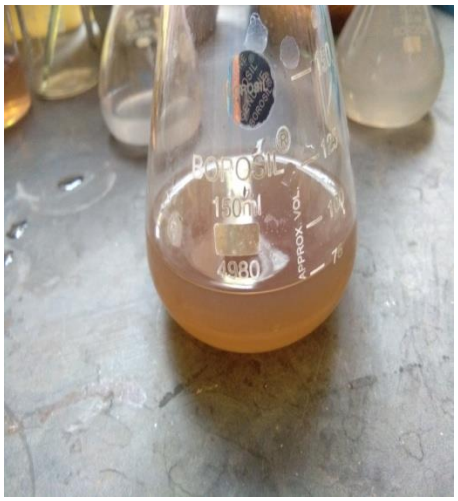


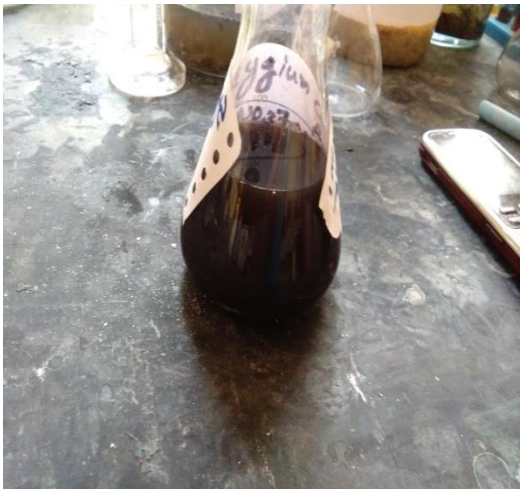


CHARACTERIZATION OF SILVER NANO PARTICLES

1. **By color change:**

The color change in reaction mixture was recorded through visual observation. The color change of the supernatant from dark purple coloured to drack browncolour indicated that the silver nano particles were synthesized.





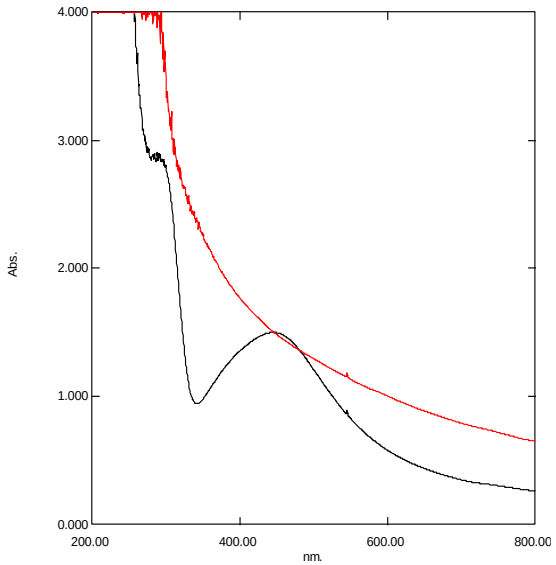
1. By color change:

The sequential color change indicates the formation of AgNPS by plant materials this is the (primary) test for the checking of formation of AgNPS. The color reduction of $AgNO_3$ into nano particles was visibly evident from the color change.Pure filter aqueous plant extract was added into a 1mM $AgNo_3$NP silver nitrate solution and boiled on water bath few minutes, the dark purpl color was changed from dark brown after 30-35 min. After 18-24 hours color changed into dark brown this color change indicate the formation of AgNPS.

2.UV Visible Spectroscopy Analysing AgNPS:

The mixture turned into dark brown colored after 18-24 hours indicating the bio transformation of ionic silver reduced to silver nano, as a result of the surface Plasmon resonance phenomenon(SPR).

The UV spectrum of AgNPS Peaks wave length at(444.50nm,342.000nm,279.50nm) The peaks were observed that was identified to be AgNPS. Generally, it is well known that AgNPS exhibit dark brown the color change ensured as of the active molecules present in the fruit extract that owing to the excitation of SPR effect. The synthesized silver nano particles were there after analyzed at different time intervals to find the stability of the particles. In this present work AgNPS were analyzed in UV visible spectrum.

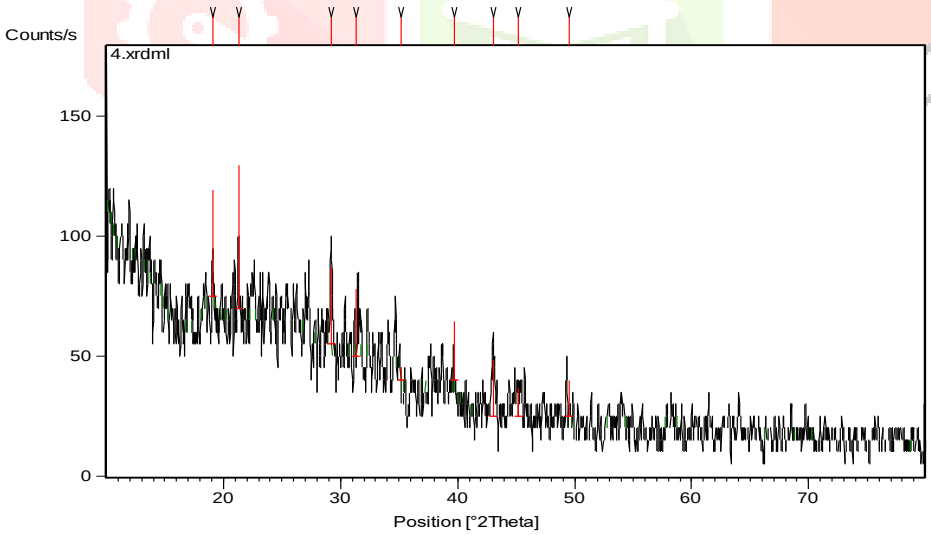


No.	P/V	Wavelength nm.	Abs.
1		444.50	1.502
2		287.50	2.904
3		253.00	4.000
4		342.00	0.945
5		279.50	2.843
6		248.00	3.97

3. X-Ray Diffraction (XRD)

X-Ray diffraction is a very important method to characterize the structure of crystalline materials and used for the lattice parameters analysis of single crystals, or the phase, texture (or) even stress analysis of sample. X-Ray diffraction of the silver nanoparticles formed from aqueous opuntia dillenii extract showed a diffraction peak 19.0500 corresponding to nanosilver.

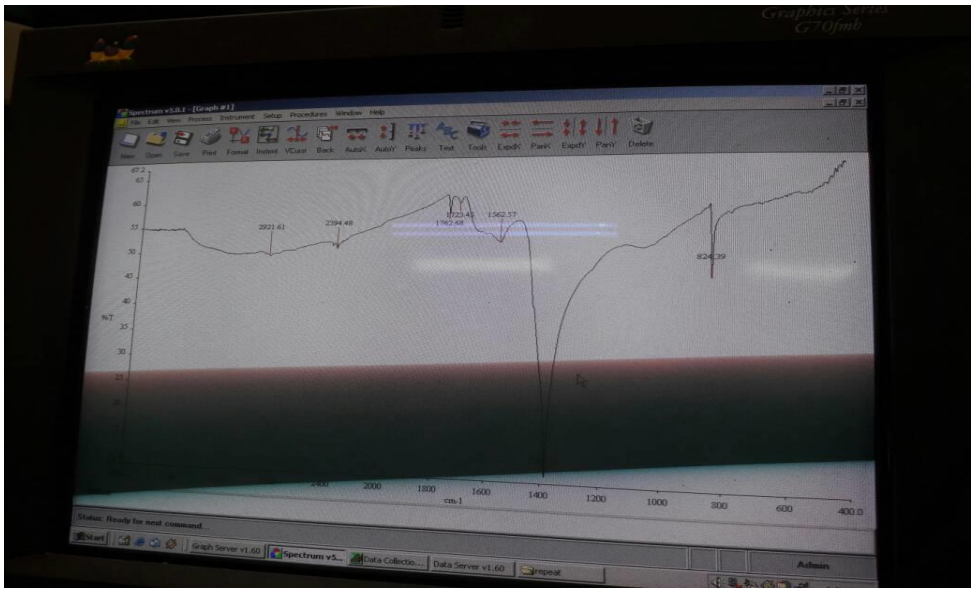
According to JCPDS standards of XRD of silver nanoparticles, the most intense peaks are related to '2θ' values of 19.0500,21.2854,29.1763,31.3500 The size of the nanoprticles was calculated by Debye Scherer's equation using FMWHS obtained from the diffraction peaks. The calculated average value for the size of the silver nanoparticles is about 112.26nm,413.77nm,4,271.9nm



Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
19.0500	8.90	0.0200	4.65885	74.21
21.2854	12.00	0.2143	4.17436	100.00
29.1763	6.48	0.4723	3.06087	54.04
31.3500	5.60	0.0200	2.85342	46.67
35.2100	1.10	0.3206	2.54895	9.17
39.7076	4.94	0.2669	2.26999	41.19
43.0742	4.75	0.4723	2.10005	39.62
45.1430	2.40	1.1520	2.00684	19.98
49.5573	3.01	0.2308	1.83944	25.10

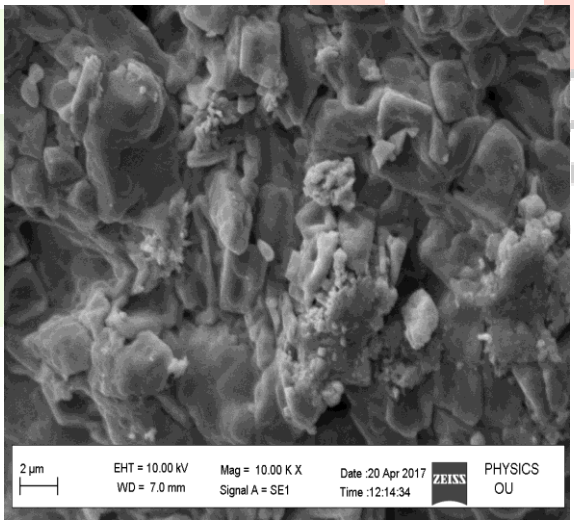
4. Fourier Transforms Infra-Red spectroscopy(FT-IR) Analysis:-

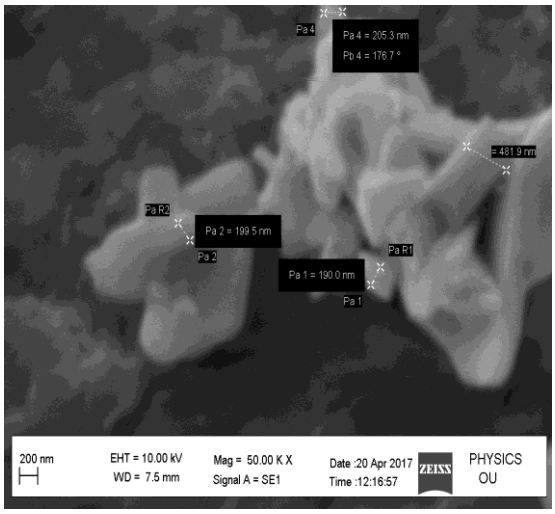
FT IR is used to prediet the rote of bonding (stabilizing)and redueing capability of opuntia dillenii fruit extract the nature of the biomolecules involved in the reduction and formation of silver nanoparticae was studied by F^{T}IR figure-2 the FTIR signals of AG-NP were observed 2921.61cm^{-1},2294.8bcm^{-1},1723.45cm^{-1},1762.58cm^{-1},1562.57cm,1384.55cm^{-1},824.45cm^{-1} the prominent bands at 2294.48cm^{-1} NH^{+} stretching due to the presence Tertiary amine saits.Which is responsible for the reduction of $AgNO_3$ to Ag and the band at 2921.61cm^{-1} may be due to the stretching of C-Hgroup the peaks at 1723.45cm^{-1},1762.58cm^{-1} due to C=O stretching vibrations of α-diketone and peak at 1562.57cm^{-1} shows that plant extract contains secondary-CO-NH amide. The Broad peak at 1384.55cm^{-1} assigned to -O-CO-CH and also due to c-h bending vibration s.the peak at 824.39cm^{-1} shows C-H- of out of plane bending of 2adj" H" atoms of β-substitated naphthalene rings the funetional bio-melecules Tertiary a mines and diketones and amide P-T-O groups involved in the reduction of silver ions,as conformed by FTIR spectrum.



5. Scanning Electron Microscopy(SEM):

Figures shows representative SEM images of the Ag nano particles synthesized by treating $AgNO_3$ solution with plant extract the resulting AgNPS were predominantly cubic pa_1=190.0nm,pa_2=199.5nm,pa_3=481nm size range from the SEM analysis of Ag nano particles from opuntia dillenii fruit supports the results. Also the rapid biosynthesis of silver nano particles of different shapes were observed and the sizes of nano particles were increased by high concentration ofopuntia dillenii fruit extract.





Conclusion:

This present investigation shows the bioreduction of silver nano particles using the medicinal valuable fruit pulp extract of opuntia dillenii fruit by optimizing the factors for rapid and stabilized nanoparticles synthesis. Nanoparticles synthesis process was controlled by incubation time, metal ion concentration and temperature. High temperature influence the rapid synthesis of silver nanoparticles.

The plant mediated method for silver nanoparticles synthesis is a very good eco-friendly method and also a non-toxic the plant sample contains naturally occurring secondary metabolites are reducing agent for the synthesis of silver metal nano particles for these characterization UV-visible spectroscopy,FT-IR,XRD and SEM were used.The colour change of the solution indicates the formation of silver nanoparticles the reduced silver nanoparticles were monitoured in UV-visible spectrophotometer at different wave lengths that is showed characteristic absorption peak at 444.50nm,342.00nm,279.500nm.

The functional groups of biomplecules are 2^0 amine groups and 1762.58cm^{-1} C=O stretching indicates presence of 4- ring membered ketone groups may responsible for the reduction process revealed by FTIR Analysis.The crystalline nature of the silver nano-particles was charactarised by X-ray diffraction and also XRD shows that structure of silver is polynoninal type of cubic and shape and size=413.77nm and 4,271.9nm.1.4482nm,112.26nm.

SEM:S anning electron microscopy(SEM)image shows figure-4 the morphological character of silver nanoparticles synthesized by using extract opunita dillenii fruit this image shows that the size at around Pa=190.0nm and pa_2=199.55nm,pa_3=481. 9nm with many shapes which are rod, and spherical are clearly observed. This SEM image also showed the aggregation of the silver nanoparticles.

This green method is simple, rapid eco-friendly,and reliable and it may have a potential use in the bio medical applications. In the future, selection of such plants may creat a new plant form for realizing t he potential of herbal medicines in nano-silence for drug delivery.

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