A novel approach to synthesis and characterization of silver nanoparticles of feverfew seeds

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ABSTRACT

Feverfew (Tanacetum parthenium) belonging to the family of Asteraceae has a great quality of curing variety of disorders. Those include migraine headaches, asthma, cancer, painful menstrual cramps, rheumatoid arthritis and many other ill complications. The seeds of feverfew (Tanacetum parthenium) were got through online from Assam, India. The obtained seeds were crushed using ethanol and the extract with 1mM silver nitrate in the ratio 1:4 was used for synthesis of Nanoparticles under agitation at 60°C. The colour change to red was detected and the further analysis with UV spectrophotometer, FESEM and X-RAY Diffractometer were done for characterizing the Nanoparticles. Red colour Nanoparticles yielded highest absorbance at 400nm followed by the particle size range at FESEM was between 12-35nm showing X-ray diffraction as FCC silver crystals at planes (111) and (200) at 36.183° and 46.233°. Thus, the silver Nanoparticles are synthesised and characterized for the further use of nano drug delivery or for the genetic analysis in curing the diseases.

Keywords: Feverfew, Silver Nanoparticles, Migraine, Cancer, X-ray diffraction, FESEM, FCC silver crystals.

INTRODUCTION

Feverfew (Tanacetum parthenium), native to Balkan Peninsula is an herb belonging to Asteraceae family having the ability to cure range of diseases from asthma to cancer. (Anil Pareek et.al.) Nano technology is a rapidly increasing field of research and wherein silver Nanoparticles play a greater role in drug delivery and in faster targeting and curing of diseases (Koushik roy et.al.). Biochemical synthesis of Silver Nanoparticles are the best ones to be used as far as the chemicals down create harm to the cells that are been injected to. This Synthesis ensures that it may also be carried out in an organism (Nair et.al.). Numerous researches have been done on the synthesis of Nano particles from biological system of for their application in the field of biomedical, pharmaceutical, cosmetic and environmental use (Shah et.al.). Although chemical & physical methods are very successful to produce well-defined Nano particles, they have certain limitations such as increase cost of production, release of hazardous by-products, long time for synthesis and difficulty in purification (Forough et.al.). The size and shape of atomic clusters or Nano particles and their ordering in 1D, 2D, or 3D arrays is to be known. In particular, silver Nano particles have been used with promising results as bactericides, anti micotics, and anticancer agents. Several methods have been devised in order to prepare metallic Nano particles (Iravani et.al.). Green synthesis of Nanoparticles paves way for economically low cost products and drugs to be manufactured from the daily used plants such that as the ancient way of treating diseases the modern medicine can also cure it using phytomedicine system and using nanodrugs produced from plant or plant extracts. (Mathur et.al., Paulkumar et.al., Abhishek et.al., Sulaiman et.al., Khalil et.al.)
EXPERIMENTAL SECTION

Preparation of Plant extract:
The Feverfew seeds were cleaned with distilled water twice and then about 100 seeds (approx. 0.5g) were crushed by addition of ethanol. Nearly 5ml of the ethanol was added to the crushed seeds and the extract is filtered using Whatman’s No.1 filter paper.

Preparation of Silver nitrate solution:
Analytical grade silver nitrate (AgNO3), a costly chemical, was purchased from local laboratory chemical suppliers. AgNO3 (100ml) was prepared to the concentration of 1mM. Weighed amount of AgNO3 (0.016g/100ml) was carefully transferred in a 100-ml volumetric flask and de-ionized water was added drop-wise while swirling to dissolve the salt up to the mark. The solution was diluted as required and all the solutions were kept away from light (the solution was changed to the brown bottle) and kept in dark.

Synthesis of Silver Nano particles:
1:4 ratios of seed extract and silver nitrate solution was taken. This was added to a beaker and heated at 60°C for 10-15minutes with agitation at 400rpm in a magnetic stirrer. This showed a colour change of colourless to red colour in those 15 minutes. This confirmed the presence of synthesized silver Nano particles in the mixture as per the protocol of Koushik Roy et.al, 2012. Then the aqueous solution was characterized using UV-visible spectrophotometer.
Characterization of synthesized Nano particles:

UV-Vis Spectroscopy:
UV-Vis spectroscopy can be comprehended as absorption spectroscopy in the spectral region of ultra-violet and visible spectra. The UV-Vis readings were taken from spectrophotometer obtained from Roy instruments in the range of 350-450nm (Birla et.al.) and observed a high peak range at ≈ 400nm and the graph is drawn using MS-EXCEL.

Powder form of the sample:
The samples were dried at 100°C and powdered.

FESEM:
FESEM (Field Emission Scanning Electron Microscope) that works with electrons (particles with a negative charge) instead of light. The FESEM analysis for the size, shape and distribution was done with the equipment seen below. It yielded results which helped in the estimation of the Nano particle size and the confirmation that the synthesized particles are present in the Nanometer range of 1-100nm with uniform distribution. The particle size was obtained exactly using software called ImageJ.

XRD:
X-ray diffraction, a common technique to study the crystalline structure of Nanoparticles was carried out in Nano research centre, SRM university where the accurate results of the crystal structure of Nanoparticles and the d-spacing was estimated using Bragg’s law and the particle size was done using Debye-Scherer’s formula (Bykkam et.al.). Finally this gave a clear result on the crystalline structure of the Nanoparticles.

EDAX:
EDAX (Energy Dispersive Absorption X-ray Spectroscopy) was done to know the elemental composition of Nanoparticles including the elements present in the seeds was studied.

RESULTS AND DISCUSSION

Synthesis of Silver Nano particles:

Fig. 2a Mixture of Seed extract and Silver nitrate
After 20 minutes, distinct change in the color of experimental samples to light wine-red was observed. Change in colour in the experimental samples clearly indicated formation of silver Nanoparticles (Figs. 2a and 2b). Undoubtedly, reports confirm similar hue of colour change due to silver Nanoparticle formation.

**UV-Vis Spectroscopy:**
After addition of seed (*Tanacetum Parthenium*) extract to the aqueous solution of AgNO₃, the mixture showed a gradual change in color at 60°C from yellowish to wine-red. The color was characteristic of the surface plasmon resonance (SPR) of silver Nanoparticles. The reduction of silver ion to silver Nanoparticle was reflected in spectral data obtained by using a UV-Vis spectrophotometer. It shows an absorbance peak around 400 nm, which was specific for silver Nanoparticles (Fig. 3). Interestingly, even other reports stating the highest absorbance in the range between 350-450 nm which clearly means that the silver Nanoparticles are present in this range and the highest absorbance confirms it is silver.
Fig. 4 X-Ray Diffraction analysis shows two peaks assigned for (111) and (200) planes of silver

X-Ray Diffraction:
XRD analysis of the prepared sample of silver Nanoparticles was done by using CuKα radiation (λ = 1.5418 Å), 2θ/θ Scanning mode, Fixed Monochromator. The XRD curve (Fig. 4) confirmed that the Nanoparticles are nothing but silver. Interpretation of this XRD pattern reveals the existence of diffraction lines at low angles (4.5° to 101°) with a step of 0.02 degree. The highest peaks were observed at the range of 35°-52°. Moreover, the high intense peak for cubic materials is generally (111) reflection, which was observed in the sample. Two peaks at 2θ values of 38.183°, 46.233° corresponding to (111), (200) plane of silver were observed and compared with the standard powder diffraction card of JCPDS, silver file No. 04-0783. From this study, considering the peaks, average particle size had been estimated by using Debye-Scherrer formula. $D = \frac{0.94}{\beta \cos \theta}$ Where “λ” is wave length of X-ray (0.1541 nm), “β” is FWHM (full width at half maximum), “θ” is the diffraction angle and “D” is particle diameter size. The particle size obtained was less than 30 nm. The value of d (the interplanar spacing between the atoms) was calculated using Bragg’s Law $2d\sin \theta = n\lambda$; $d = \frac{\lambda}{2\sin \theta}$ ($n = 1$) Wavelength $\lambda = 1.5148 \text{ Å}$ for CuKα

<table>
<thead>
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<th>2θ of the intense peak (deg)</th>
<th>hkl</th>
<th>θ of the intense peak (deg)</th>
<th>FWHM of intense peak (β) radians</th>
<th>Size of the particle (D) nm d-spacing nm</th>
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<tr>
<td>38.183</td>
<td>(111)</td>
<td>19.09</td>
<td>0.0104</td>
<td>0.2366</td>
</tr>
<tr>
<td>46.233</td>
<td>(200)</td>
<td>23.126</td>
<td>0.0087</td>
<td>0.1961</td>
</tr>
</tbody>
</table>

Fig. 5a FESEM image at 1µm width in high vacuum
FESEM (Field Emission Scanning Electron Microscopy):
Field Emission Scanning Electron Microscopy was performed for revealing the surface morphology, distribution and density of Nano particles. The above images (Fig.5a, 5b, 5c) were obtained and it was found that the Nanoparticles were uniformly distributed and were denser with Nano particle size ranging between 12-35nm. The
software Image J was used to find the exact particle size. The size range of 30-100nm of the silver Nanoparticles fit in perfectly for the drug delivery on the cells. Interestingly, our particle had an intermediate range in this.

**EDAX (Energy Dispersive Analysis of X-Ray):**

Energy Dispersive X-ray Spectroscopy or EDAX, a technique, mainly used to identify the presence of different elements in a sample. In the present study, EDAX shows the presence of Ag, C, O, Mg, Na, Ca, and Si and the curve (Fig.6) shows the peaks of all the above elements. The presence of other elements along with Ag was due to their prevalence in the seeds. This confirms that the seeds are rich in nutrients. The synthesized Nanoparticles contain 0.56% of silver by weight.

**SUMMARY:**

- The silver Nano particles were synthesized and the colour change from pale yellow to red was observed.
- UV-Vis spectrophotometer readings were taken at the highest absorbance was noted at 400nm.
- The samples were dried and powdered at 100°C.
- X-ray diffraction spectrum was obtained using Powdl converter.
- The Pattern analysis was done and the respective Particle size, Miller indices (hkl) values and the intense peak analysis was done and the values were obtained successfully.
- FESEM analysis was done and it was found that the particles were uniformly distributed, ranging from 12-35nm particle size.
- EDAX analysis was done and found that elements like C, O, Mg, Na, Ca, and Si were present in addition to Ag Nano particles which were synthesized.

**CONCLUSION**

The above study confirms the synthesis of Nanoparticles from the Tanacetum parthenium seeds. This also emphasizes the crystal form of the silver Nanoparticles which is cubical. The Nanoparticles size ranges between 12-35nm. This study clearly depicts the quality of feverfew seeds on its ability to synthesize silver Nanoparticles and its nature to have its rich nutrients on the synthesized Nanoparticles. Thus, the study helps in the clear picture of Nanoparticle synthesis and characterization using the feverfew seeds.

**FUTURE WORK PLAN:**

This work had been planned to be extended further with drug discovery and drug delivery using the synthesized silver Nano particles. Since silver has the quality of entering the cell easily, the silver nano drugs may be useful to cure the diseases like migraine, cancer, and other neurological disorders. The molecular level study could be done and the genetic analysis and gene expression of the *Tanacetum parthenium* over the human cell lines can also be understood.

**Acknowledgement**

I hereby thank Bharath University, my department staff members for extending their support and encouragement to complete this project. I thank NRC, SRM University, Chennai for providing me results accurate and on time. I thank Dr. Balakrishnan, Department of Nanotechnology for his help on working out the XRD data. I also thank my parents for providing me funds for analyzing data.

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