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Banana leaves mediated bio-synthesis of silver nanoparticles

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Silver (Ag) ions are successfully reduced to stable silver nanoparticles (AgNPs) using banana leaves. Conversion of Ag ions to AgNPs has been confirmed by UV-VIS analysis. The result of change on independent variables, i.e. temperature and strength of reducing agent on absorbance of solution and optimization of parameters has been done using response surface methodology (RSM) as per central composite design (CCD). The type of chemical group present on the nanoparticles is studied using fourier-transform infrared spectroscopy (FT-IR). Estimation of the size of particles has been done using transmission electron microscopy (TEM) and nanoparticle size analyzer. The crystallinity of the nanoparticles formed has been confirmed using XRD. Energy dispersive X-ray analysis (EDX) data is used to confirm the nanometal present.

Keywords: Banana, Leaves, Nanoparticles, Response surface methodology, Silver.

Metal nanoparticles (NPs) display peculiar optical, electrical and catalytic properties which can be tuned by controlling their size and shape[1-3]. Therefore, a huge quantum of exploration has been devoted to regulate the morphology and dimension of metal NPs. The methods developed to prepare and synthesize these NPs include a variety of physical and reduction techniques such as chemical[4-7], electrochemical[8,9], photochemical[10,11]and heat evaporation[12,13], etc.

The discovery of the fact that AgNPs can be used to obtain antimicrobial effects has led to the incorporation of nano silver into several consumer products such as clothing and cosmetics. Inspite of several benefits of these nanoparticles, their release into the environment is a cause for concern to environmental biologists around the world[14].

Chemical synthesis, by reduction of metal ions to metal nanoparticles, use toxic chemicals as reducing agents and capping agents[15,16]. Hence there is a need for green synthesis of metal nanoparticles which does not employ toxic chemicals and thereby the environment is protected[17]. This can be achieved by utilizing products and materials from various plant sources as well as other biological materials as reagents in the synthesis protocol. More and more research inputs are rendered in this direction and the synthesis of nano materials is increasingly being carried out by using several plants, plant extracts, biochemicals from plants and micro-organisms[15,18-21].

In this work, Response Surface Methodology (RSM) was employed to monitor the effect of major process variables such as temperature and reducing agent strength on the synthesis of AgNPs by using a reducing agent from a biological source, that is, banana leaves, to provide a green route to NPs synthesis. Synthesized NPs were then characterized using different techniques.

Experimental Section

Materials

Silver nitrate $AgNO_3$ (Mol. Wt. 169.87g) and acetic acid analytical grade were purchased from S. D. Fine-Chem Ltd (SDFCL), Mumbai, India.

Synthesis of AgNPs

Banana leaves were collected from the campus, washed with deionized water and cut into small pieces. 0.01 M aqueous solution of silver nitrate of 100 mL was prepared in a conical flask using distilled water, the pH was then adjusted to 3 with the help of acetic acid. In this salt solution, banana leaves were added with continuous stirring on a shaker bath machine (RossariLabtech, Mumbai, India) at 70 rpm for two hours. Experiments were conducted according to the Design of Expert 7 to study the result of variation of two parameters, temperature and concentration of banana leaves which behave as reducing agent. Reduction of silver ions into AgNPs was observed from change in color (and hence UV absorbance) of the solution from colorless to reddish brown, confirming the formation of NPs[22].The solution was filtered through a nylon mesh with suitable pore size, after complete reduction, centrifuged at 12000 rpm for 15 min, washed with distilled water and allowed to dry at 80°C.

Table 1— CCD experimental run of trials for synthesis of AgNPs

Run	Temperature (° C)	Concentration of Reducing agent (gm/ 100 ml)	Absorbance at 429 nm	
			Experimental	Predicted
1	100	7.5	0.5804	0.58574
2	100	2.5	0.1932	0.192773
3	50	2.5	0.1822	0.16694
4	100	5	0.3952	0.390287
5	50	7.5	0.344	0.334506
6	75	5	0.3673	0.344452
7	50	5	0.227	0.251754
8	75	5	0.3392	0.344452
9	75	2.5	0.1876	0.203287
10	75	7.5	0.4794	0.483554
11	75	5	0.3794	0.344452
12	75	5	0.3138	0.344452
13	75	5	0.3424	0.344452

Experimental design

Experimental parameter optimization was done using RSM as per central composite design(CCD) to study the result of variation of independent experimental parameters, i.e. temperature and concentration (or strength) of banana leaves on the UV-Vis absorbance of the solution after each experimental run as a response which is displayed in Table 1.

Characterization

A UV-Visible spectrophotometer (UV-1800 ENG 240 V, Shimadzu, Japan) was used for analysis of synthesized AgNPs measured as a function of time in the UV-Visible range. The particle size was determined using a nano particle size analyzer (SALD 7500 nano, Shimadzu, Japan). Surface morphology was elucidated by transmission electron microscopy (Phillips TEM-200 Supertwin STEM, accelerating voltage-200kV, resolution-0.23 nm). Crystallographic study of nanoparticles was conducted using an X-ray diffractometer (Shimadzu XRD-6100, Japan) withCuKα radiation 40kV/30mA using the 2θ range of 10–80º. Chemical functional groups on AgNPs were identified using FTIR (FTIR 8400S Shimadzu, Japan) with the spectral range of 750-4000 cm^{-1} and elemental analysis was done using EDAX (EDX-720, Shimadzu, Japan).

Results and Discussion

Optimization analysis

To elucidate the correlation between the response (in terms of spectrophotometric absorbance) and independent variables and to determine the maximum yield of AgNPs by the present green synthesis procedure, corresponding to the optimal values of temperature (A) and concentration of banana leaves (B), a second-order polynomial model was envisaged to calculate the optimized values of the variables. The results of experiments are presented in Table 1.

Table 2 — Analysis of Variance (ANOVA)

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	0.161149	5	0.03223	56.87586	0.0001
Pure error	0.002636	4	0.000659		
R^2	0.9760				
Adj. R^2	95.88%				

ANOVA results are presented in Table 2. The p-values isemployed to evaluate the significance of each coefficient which is also an indication of the interaction strength of each parameter. For the present work, the F-value (56.87586) and p-values (p = 0.0001) indicated statistical significance of the obtained model. It can be seen from the degree of significance that the quadratic effects of temperature and concentration of banana leaves which acted as reducing agent are quite significant, meaning that they were the deciding factors and small variation in its value will affect the amount nano particles produced. The value of adjusted-R^2 = 0.9588indicated that the total variation of 95.88% of absorbance is attributed to the independent variables and only 4.12% cannot be explained by the model[23].

After correlating independent and dependent variables at optimized temperature and reducing agent concentration to yield maximum AgNPs, a second-order polynomial model was obtained (Equation 1). This equation was obtained by applying multiple regression analysis on experimental data.

$$\text{Absorbance} = -0.020518 + .00388611A - 0.00991701 B + .000901600\,AB - 0.0000374897\,A^2 - 0.00001649B^2 \quad \cdots(1)$$

As the temperature and concentration of reducing agent increase, absorbance of synthesized nanoparticles increases (Fig. 1) which indicates increase in concentration of AgNPs. According to the RSM, the results predicted by the model showed that the maximum absorbance could be achieved when the temperature and concentration of reducing agent were set at 64.42°C and 4.87g/ 100 mL respectively. The maximum predicted value of absorbance obtained was 0.304869. Under these optimized conditions, the mean experimental value of the absorbance was 0.3028, which was near about the predicted value.

Characterization

UV- Visible spectral analysis

The UV-visible spectra of AgNPs with increasing reaction time are shown in Fig. 2. The absorbance peak seen in the region of 425-475 nm indicates the presence of AgNPs due to surface plasmon resonance[22].

Figure 3 shows, the reduction of silver nitrate into AgNPs which was observed by change in color of solution from colorless to reddish brown with increasing reaction time[22].

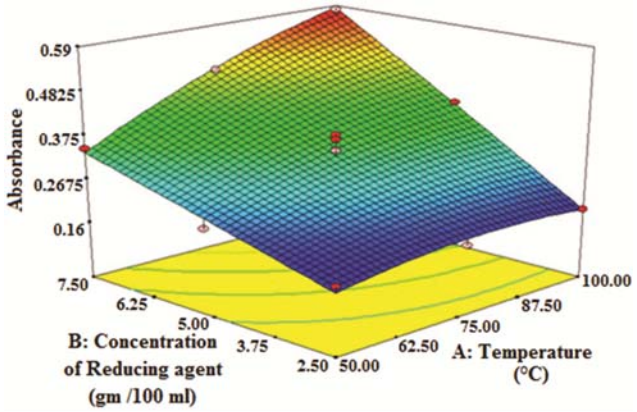

Fig. 1 — Response surface plot of Absorbance Vs Temperature and Concentration of reducing agent.

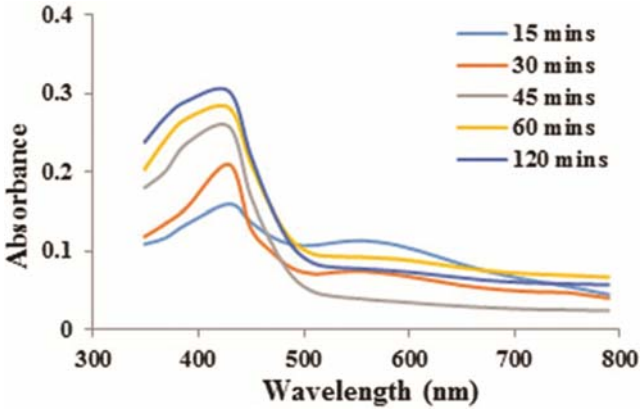

Fig. 2 — UV-Vis absorption spectra of AgNP solutions at different reaction times, minutes.

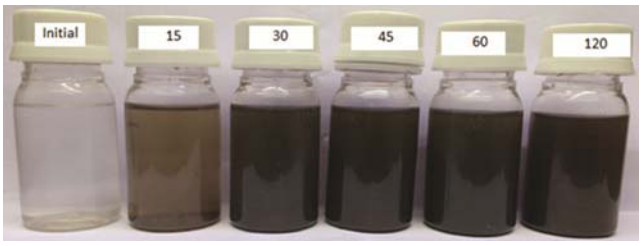

Fig. 3 — Samples of AgNPs solutions taken out at different time intervals in minutes.

Particle size analysis and morphology of AgNPs

Figure 4 shows the particle size analysis of AgNPs having average particle size of 40 nm. The TEM image in Fig. 5 shows that the AgNPs were mostly having spherical geometry. It also shows that the particles varied in size from 2 nm to 12 nm having an average diameter of 8 nm.

X-Ray Diffraction analysis

Figure 6 shows the XRD pattern of AgNPs having four peaks at 37.6891°, 43.8200°, 64.0908° and

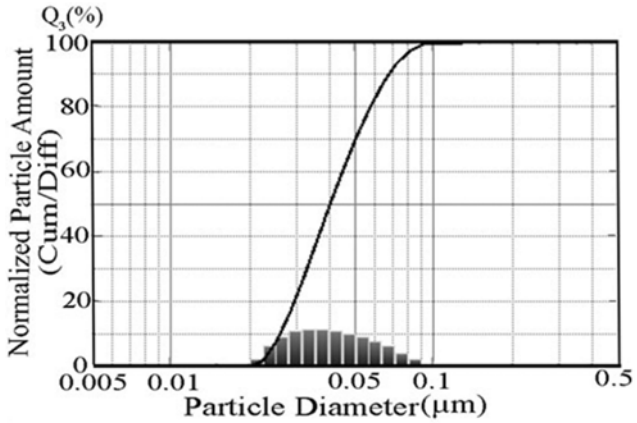

Fig. 4 — Particle size distribution of AgNPs.

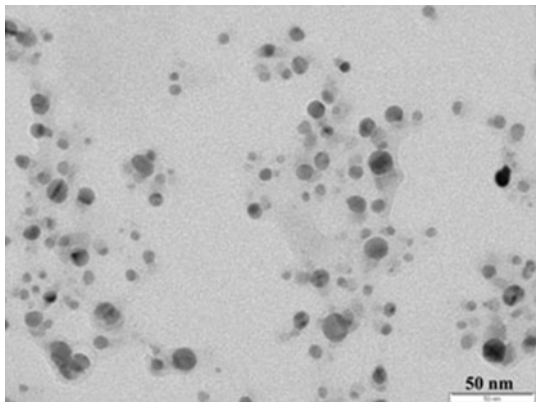

Fig. 5 — TEM image of AgNPs

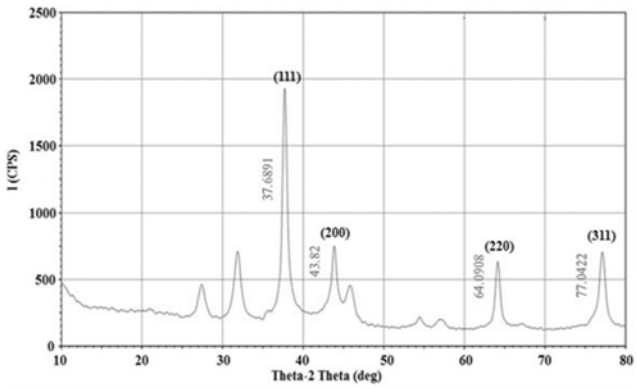

Fig. 6 — XRD pattern of AgNPs

Table 3 — Particle size of Ag nanoparticles calculated by different methods

Crystallite size (nm)		
Debey-Scherrer formula	Dynamic Light Scattering technique	TEM
12.72	40	8

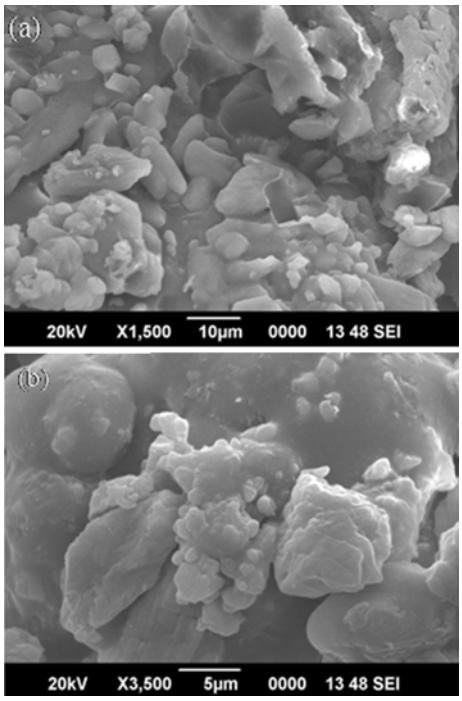

Fig. 7 — EDX pattern of AgNPs

77.0422° matching with (111), (200), (220) and (311) Miller indices respectively. The intensity of the pattern clearly established the formation of AgNPs (JCPDS card no 04-0783, 1991). It showed crystallinity of 38.8257% as is proven by the sharpness of the (111) peak[24].

Particle size calculation

The average particle size was calculated by Debey–Scherrer's formula

$$D = \frac{0.94\lambda}{\beta \cos e} \quad \ldots (2)$$

where β is full-width at half maximum (FWHM) of the diffraction peak in radians, λ is X-ray wavelength (0.15406 nm) and Θ is Bragg's diffraction angle respectively. The particle size obtained was 12.72 nm[25].

The AgNPs size as deduced by above methods aredisplayed in Table 3.

Energy Dispersive X-Ray analysis

The absorption signal in the range of 3 to 4 keV is characteristic for the absorption of metallic silver nanocrystallites was obtained for the synthesize AgNPs (Fig. 7) confirming its presence[26].

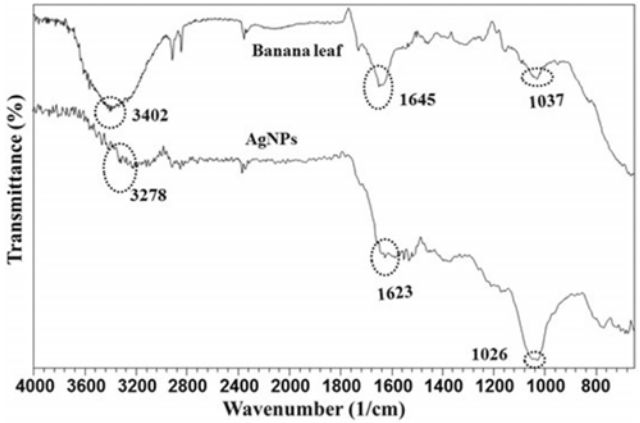

Fig. 8 — FTIR spectrum of Banana leaves and AgNPs

FTIR analysis

Figure 8 shows the FTIR spectra of banana leaves and the synthesized silver NPs. The O–H stretching around 3400 cm^{-1} shows the presence of hydroxyl groups from the polyols such as flavones, terpenoids and polysaccharides present in the leaf extract. The bands at 1645 cm^{-1} and 1037 cm^{-1} denote the C–N, C–O stretching from organic material in the banana leaf extract[27].The decrease in intensity of bands as well as the considerable broadening might be due to interaction with nanoparticles. These bands interpret the presence of compounds like flavonoids, polysacharides and terpenoids might be the reason for stabilization and capping of the AgNPs[28].

Conclusion

AgNPs have been successfully prepared by using banana leaves as reducing agent. The presence of flavonoids, terpenoids and proteins in banana leaves are responsible for reduction of silver ions from silver nitrate into AgNPs. These compounds are also present on the surface of nanoparticles, providing stability to the colloidal particles. The formation of nanoparticles gives reddish brown solution giving an absorbance peak in the range of 425-475 nm in the UV-visible spectroscopy analysis. For maximum absorbance (0.0304869) the optimized conditions were 64.42˚C temperature and 4.87 gm/100mL concentration of reducing agent as per CCD. The AgNPs formed were of predominantly spherical shape and crystalline nature with crystallinity of 38.8257%. The particle size of AgNPs was 31 nm, 61.94nm and 72nm observed respectively with particle size analyzer, XRD and TEM.

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Competing interests section

The authors declare that they have no competing interests.

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