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Ultrasound Intensified Green Synthesis of Silver Nano particles using Camellia Sinensis Extract fortified with Lemon and Honey

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Abstract: Ultrasonic waves appear as an interesting way to improve process productivity.The present study reports a facileultrasonicsintensified greensynthesis of silver nano particles from the extract of Camellia Sinensisfortified with lemon and honey. The extract is acting both as reducing agentas well as capping agent.The synthesized silver nano particles have been characterized by UV-Vis spectroscopy, Scanning Electron Microscopy(SEM) and Energy Dispersive X-ray (EDX) Analysis. The active organic groups responsible for the reduction of Ag^+ ions to elemental silver were identified by Fourier Transform Infrared spectroscopy(FT-IR).The effect of ultrasound on the reduction reaction rate was found andwas found to get enhanced with ultrasonic irradiation.Polydispersity of synthesized silver nano particles is minimized and alsocontrolled by ultrasonication method. This novel method is rapid, facile and combines the advantage of both sonochemistry and green chemistry.
Key Words: Green synthesis; process intensification; ultrasound assisted synthesis; Bio-reduction.

Introduction

Metal nano particles exhibit distinct physical and chemical properties compared to their bulk counter parts due to the small size, shapes and controlled disparity.[(1)] Silver nano particles have always attracted researchers due to its wide spread application in various fields such as catalysis, sensors,[(2)]electronics, biotechnology and in biomedicine as antibacterial,[(3)]antiseptic,antimicrobial, [(4)]antiviralagents etc.,Though various conventional methods like electro-chemical,[(5)]sol-gel process,[(6)] chemical reduction,[(7)]hydrothermal,[(8)]Sonochemical[(9)] microwave irradiation[(10)] etc.,methods are available for the synthesis of silver nano particles,in the past few decades there has been an extensive increase in the research for green synthesis[(11-20)]of silver nano particles due to environmental concerns and sustainable development which makes it an attractive alternative to the conventional methods of synthesis. Green synthesis of silver nano articles using natural sources is currently under exploitation[(21).]

Several polyphenoliccatechinsviz,(-) epicatechin(EC),(-) epicatechin-3-gallate(ECG),(-) epigallocatechin (EGC), (-) epigallocatechin-3-gallate(EGCG),(+)Catechin, and (+) gallocatechin (GC)which are present in the camelliasinensis extract are strong antioxidants and canpotentially reduce silver salts. The reducing nature of camellia sinensis was enriched with lemon(citric acid component) and honey (poly saccharide). The process is further intensified by irradiation with ultrasonic waves. When the reactants are irradiated with ultrasound the alternating expansive and compressive acoustic waves create bubbles called cavities and it make the bubbles to oscillate.[22-25].The oscillating bubbles accumulate the ultrasonic energy effectively and grows, when it becomes optimum in size , it collapses releasing the concentrated energy stored in the bubble within very short-time. The cavitational implosion is very localized and transient with a temperature of the order of thousands kelvin,that assists in the production of silver nano particles of smaller size and with reasonably faster rate.

Materials

Precursor- Silver Nitrate-purchased from Qualigens, India.Long leaves of camellia sinesis, natural honey and lemon .

Instrument: WensorDigital Ultrsonic bath

Extract preparation:

One gram of well dried and powdered Camellia Sinesiswas put in to 50ml of water and boiled for about 15 minutes.The extract was filtered withWhatman No.1 filter paper. To the extract 5ml of Honey and 5ml of lemon juice was added. This aqueous extract was used to synthesize silver nano particles.

Results and Discussion

When 5ml ofcamellinasinensis with lemon and honey extract was added to 100ml of 0.001M Silver nitrate solution, Color of the solution changed gradually from pale yellow to reddish brown colour in fifteen minutes. Appearance of Reddish brown colour indicated the formation of silver nano particles. When the same amount of the precursor and the aqueous extract was irradiated with ultrasonic waves, it has taken only five minutes of time for the appearance of reddish brown color and thereby for the formation of silver nano particles. This may be due to the fact that in addition to the active groups (present in the extract) responsible for the bio- reduction, ultrasonic irradiation of aqueous liquids generate free radicals like $H^{\cdot}$ and $OH^{\cdot}$, these free radicals[22] also reduce Ag^{+} ions to Ag(0). Hence the reduction reaction rate got enhanced with sonication.

Characterization

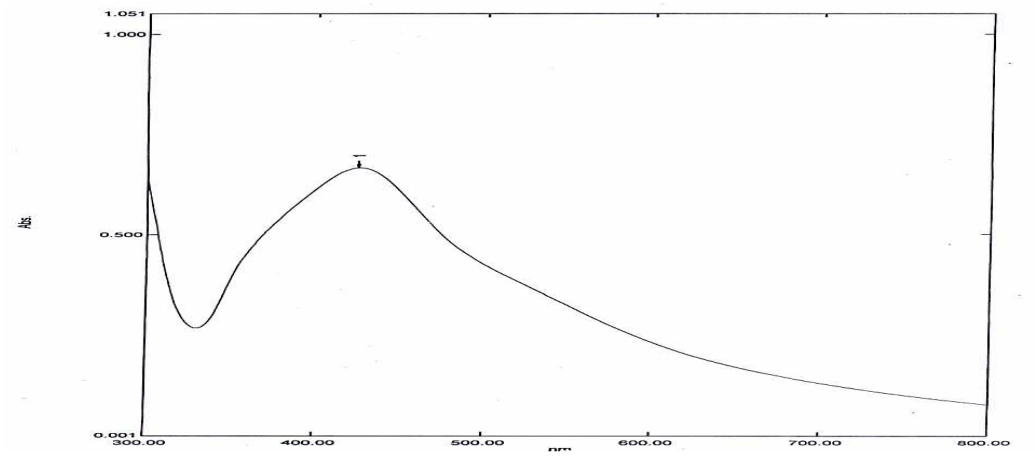
UV-Visible Spectral Analysis:

The initial characterization of the synthesized silver particles was done by UV-Vis spectroscopy.

Sample: The bio- reduced aqueous component (0.5ml) was diluted ten times with double distilled water to avoid errors due to high optical density of the solution.

Instrument: Shimadzu UV 1650pc Spectrophotometer.

Scanning Range:350nm to 600nm



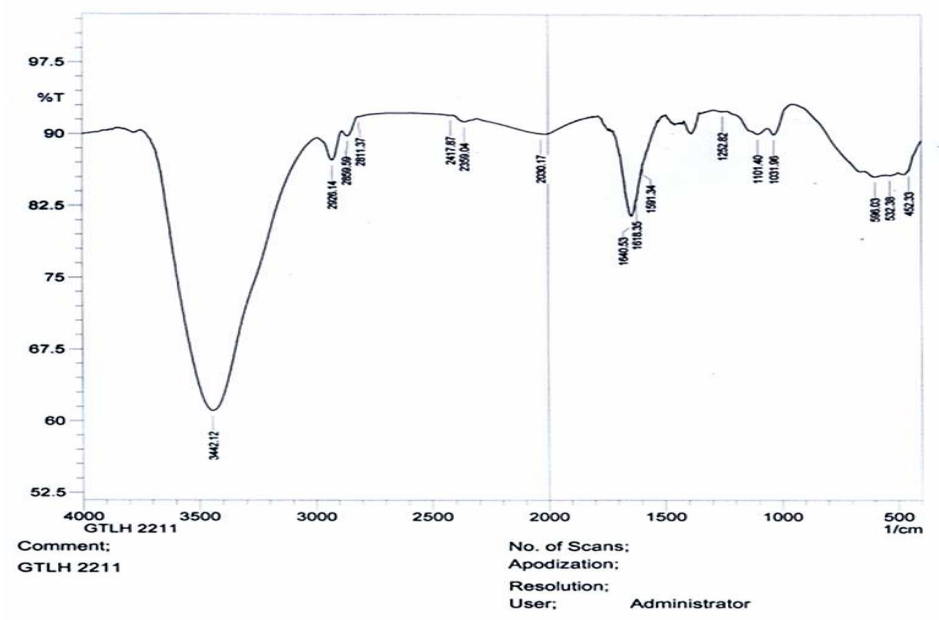
Metal nano particles have free electrons, which gives surface plasmon resonance absorption band due to the combined vibration of electrons of metal nano particles in resonance with light wave. The appearance of a sharp absorption peak at 425nm, is characteristic wavelength of silver and thereby confirms the formation of silver nano particles.

Fourier Transform Infrared Spectral Analysis (FT-IR)

Sample: The bio- reduced solution was centrifuged at 10,000 rpm for half an hour. The sample was grinded with KBrpellets , dried in infrared light and then subjected to FT-IR measurement

Instrument: Shimadzu FT-IR Spectrophotometer.

Spectrum Range:Spectrum was recorded in the range of 4000-400cm^{-1}



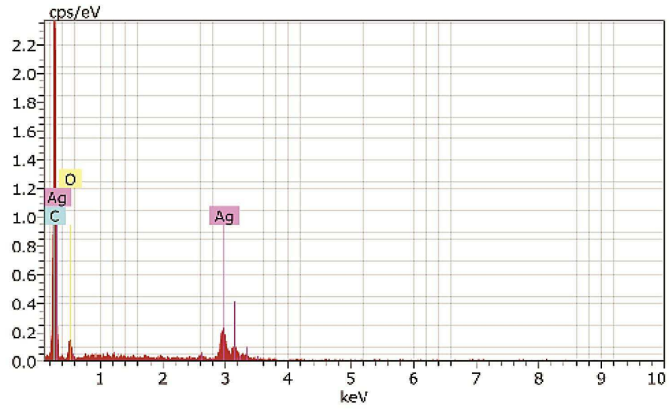
The intense peak at 3442cm^{-1} correspond to N-H, O-H stretching vibrations of alkanes, amide, alcohol and H-bonded to phenols. Peaks at 2950, 2926and at 2811 indicates the presence of very strong C-H strectching vibrations. The peak at 2080 corresponds to C=N stretching vibration. The peak at 1640 indicate C=C, C=O stretching vibrations of alkenes and amides. The peak at 1031 indicates C-O, C-N stretching vibrations and it

corresponds to the presence of alcohols, carboxylic acids, ethers, esters and aliphatic amines in the extract. The peak at 596, 532,and 452 corresponds to C-Cl, C-Br and C-F stretching vibrations of alkyl halides. So it can be assumed that the water soluble alkaloids, flavonoids and phenols present in the extract are acting as both capping agent as well as stabilizing agent.

Energy Dispersive X-Ray Analysis (EDX)

It is an analytical technique to identify the elemental composition of the specimen.

Instrument: The dried silver nanoparticles prepared were mounted on specimen stubs, and are examined under a Philips XL-30.



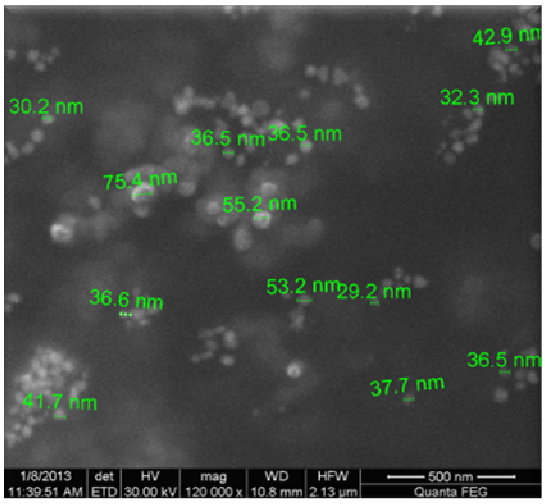
Spectrum: Acquisition

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
C	6	K-series	51.54	55.32	84.47	7.59
Ag	47	L-series	34.06	36.56	6.22	1.51
O	8	K-series	7.57	8.12	9.31	2.27
		Total:	93.17	100.00	100.00	

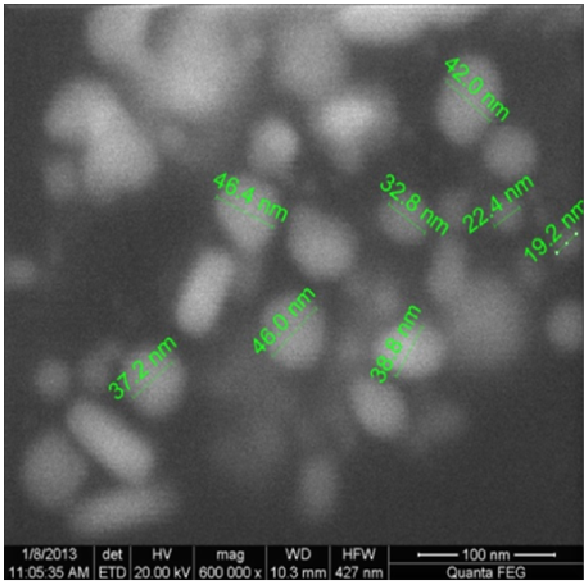
EDX attachment on the SEM provided the chemical analysis of the field as well as spot analyses of minute particles and confirmed the presence of specific elements. The presence of elemental silver in the reaction mixture was confirmed by EDX analysis. The silver nano particle exhibited an optical absorption band peak at 3 KeV which is typical of the absorption metallic silver nano particles.

SEM Analysis

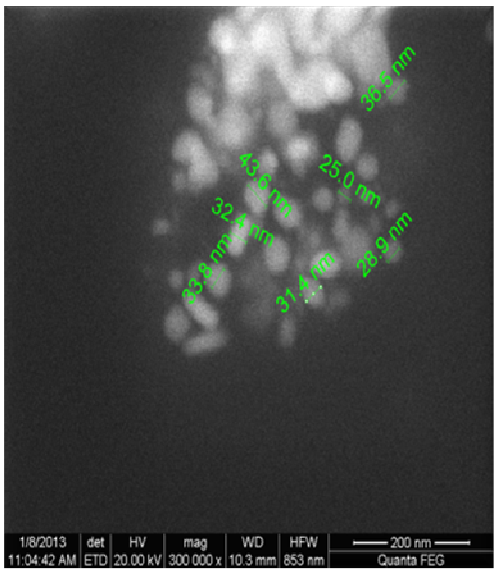
Sample: Thin film of the sample was prepared on a carbon coated grid by just dropping a very small amount of the sample on the grid, extra solution was removed using a blotting paper and then the film on the SEM grid was allowed to dry by putting it under mercury lamp for 5 minutes.



Silvernano particles formed before ultrasonication



after sonication for I hour



after sonication for 2 hours

SEM images of silver nano particles at different magnifications before and after ultrasonication reveal that the particles are predominantly spherical in shape. The average size ranges from 30.2 to 75.4 before ultra sonication. After ultra sonication, poly dispersed particle size of silver nano particles is reduced to an average size of 34.5nm and almost of uniform particle size.

Conclusion

In this work, we were able to prepare silver nano particles by a clean, safe and rapid method using all natural sources,camellia sinensis, honey and lemon extract, and the process was intensified by ultrasonication. The synthesized silver nano particles have high potential applications in the bio medical field and this procedure has several advantages such as cost effectiveness, compatibility for medical and pharmaceutical applications as well as large scale production.

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