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Research Article

One-Pot Fabrication and Characterization of Silver Nanoparticles Using *Solanum lycopersicum*: An Eco-Friendly and Potent Control Tool against Rose Aphid, *Macrosiphum rosae*

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The espousal of nanotechnology is a current come-up of the present revolution. As we know that the rose aphid, *Macrosiphum rosae* (Hemiptera: Aphididae), is a key pest on rose plant in Kashmir Valley, India, it exhibits a worldwide distribution. In the present study, we have synthesized biologically silver nanoparticles (Ag NPs) from *Solanum lycopersicum* and characterized them by UV-vis spectroscopy, TEM, and X-RD analysis. The experiment was performed by leaf dip method. Insecticidal solutions of different Ag NPs concentrations, namely, 200, 300, 400, and 500 ppm, were tested on *M. rosae*. For assessment purposes, leaves were treated with distilled water (used as control). Aphid mortality data revealed that the Ag NPs were effective at 500 ppm concentration. As the concentration and day's treatment increased, the aphid mortality rate also increased. There were statistically significant differences in *M. rosae* mortality between concentrations by LSD at 5%. In wrapping up, the use of Ag NPs in pest control processes will be the most novel eco-friendly approach in the Kashmir Valley, India, in future.

1. Introduction

Rose is a prickly ornamental shrub which is the most beautiful and attractive flower in the world in Kashmir, India, often referred to as "Queen of Flowers." The genus *Rosa* is endemic to temperate regions of the Northern Hemisphere, including North America, Europe, Asia, and Middle East [1]. Roses are used to produce rose oil, rose water, concrete, and absolute, all of which are valuable and important base material for perfume and cosmetic industries [2]. But their main use is in cut flower industry and landscaping and they are mainly used in trade. However, rose production is significantly lowered by phytophagous insects especially aphids which are most unsafe because of their large populations and kind of damage [3]. Out of the different aphid species, *Macrosiphum rosae* (Linn.) is a key pest of rose, causing direct damage by sap sucking which results in discoloration of leaves, stunted growth, and gall formation, while indirect loss is incurred by honeydew secretion on which molds grow resulting in reduction in photosynthesis and finally the yield [4].

Application of synthetic insecticide is common practice to control insect pests in South East Asia and throughout the world. A number of effective insecticides have been reported for control of rose aphid. However, this sucking insect pest associated with rose crop has been long known to develop resistance [5]. Moreover, the uses of chemical insecticides result in secondary pest outbreak [6] besides being encountered in food products, adipose tissue, and milk of animals and have harmful effect on nontarget organisms as well [7]. The situation thus demands sustainable alternatives [8]. The use of nanoparticle pesticide is one such avenue which has potential to revolutionize modern day insect pest control [9–11].

Nanoparticles possess insecticidal efficacy due to their novel assets like extraordinary strength, chemical reactivity, and electrical conductivity. They have significant property of self-assembly, stability, specificity, encapsulation, and biocompatibility [12–14]. Thus, nanotechnology has become one of the most promising new approaches for pest control in the recent years [15]. Nanotechnology represents a new generation of environmental remediation technologies that could provide cost effective solution to some of the most challenging environmental cleanup problems [16–18]. Silver has been used in many applications in pure free metal or in compound form because it possesses antimicrobial activity against pathogens, yet it is nontoxic to human in relation to its quantity [14, 19, 20]. Although there have been numerous studies on the toxicity effects of nanoparticles on bacteria, fungi, and animal pathogens [20–25], little work has been carried out to find the lethal effects of nanoparticles on insect pest control.

Tomato (*Solanum lycopersicum* L.) is one of the most important vegetables worldwide because of its high consumption, year round availability, and large content of health related components. The consumption of tomatoes has been proposed to reduce the risk of several chronic diseases such as cardiovascular diseases and cancer [26, 27]. In addition, tomato consumption leads to decreased serum lipid levels and low density lipoprotein oxidation [28]. To the best of our knowledge, the biotoxicity potential of *S. lycopersicum* against *Macrosiphum rosae* is unknown. In this research paper, we report a method to biosynthesize Ag NPs using the extract of *S. lycopersicum*, a cheap and eco-friendly material acting as reducing and stabilizing agent. Ag NPs were characterized by UV-vis spectrophotometer, X-ray diffraction (XRD), and transmission electron microscopy (TEM). The biosynthesized Ag NPs were tested for observing their mortality effects on *Macrosiphum rosae* at different concentrations.

2. Materials and Methods

2.1. Materials. Silver nitrate ($AgNO_3$) was purchased from Sigma Aldrich Chemicals, India. The glassware was acid-washed thoroughly and then rinsed with Millipore Milli-Q water. *Solanum lycopersicum* L. was collected from local supermarket in Kodaikanal, Tamil Nadu, India.

2.2. Synthesis of Silver Nanoparticles. Synthesis of Ag NPs was carried out by the method of Umadevi et al. [29], with slight modification, where 100 g of washed *Solanum lycopersicum* was compressed by the mixer grinder for extraction. The extract was estranged by centrifugation at 1000 rpm for 15 min to remove inexplicable fraction and macromolecules. Then the light yellow extract was collected for further experiments. 15 mL of *S. lycopersicum* extract was mixed to 50 mL aqueous solution of $AgNO_3$ (1 mM) and stirring continued for 2 min at 30°C. Reduction takes place rapidly as indicated by reddish brown color of the solution which gives silver colloid. During the synthesis, the prepared nanoparticles, initially, become colorless and turned into a characteristic reddish brown for Ag NPs; after that the prepared nanoparticles showed no further change in color, implying the completion of reaction. This stability results from a potential barrier that develops as a result of the competition between weak Van der Waals forces of attraction and electrostatic repulsion.

2.3. Characterization Studies. The bioreduction of Ag^+ ions was monitored using UV-vis spectrophotometer (UV-160v, Shimadzu, Japan). Analysis of size and morphology of Ag NPs was performed by transmission electron microscopy (TEM Technite 10 Philips). The purified crystalline Ag NPs were examined by XRD analysis.

2.4. Insect Culture and Bioassay. A colony of test insect, *Macrosiphum rosae,* was established on potted rose plants under a temperature range of 20–22 ± 2°C in the Entomology Laboratory, Department of Zoology, University of Kashmir, India. Bioassay for effects of the Ag NPs on wingless (apterous), viviparous female aphids was taken into account for our experiment. The experiment was determined by dipping leaves in different concentrations (200, 300, 400, and 500 ppm) which were prepared by dissolving 2, 3, 4, and 5 mgs of Ag NPs in 10 mL of distilled water followed by addition of 0.05 mL ethanol to observe the more or less clear solution. Moreover, 10 mL of distilled water with 0.05 mL ethanol was taken for control experiment. The solutions were stirred continuously for about an hour with a polished blunt end of a glass rod. The nanoparticle solution turned brownish in color and was kept as such overnight, thereafter accessible for our experiment. Then we released 10 female aphids on the treated leaves. Insect mortality was counted after 24, 48, 72, and 96 hours of treatment.

2.5. Statistical Analysis. Statistical analysis was performed using SPSS version 20.0 for Windows. The mortality data were analyzed using one-way ANOVA to compare effects among treatments. The results were expressed as percentage mortality (±SE) and treatment effects were statistically significant at $P \leq 0.05$ using LSD test.

3. Results

The Ag NPs were synthesized from *S. lycopersicum* extract by maintaining the time and then 50 mL aqueous solution of $AgNO_3$ (1 mM) and stirring continued for 2 min at specific

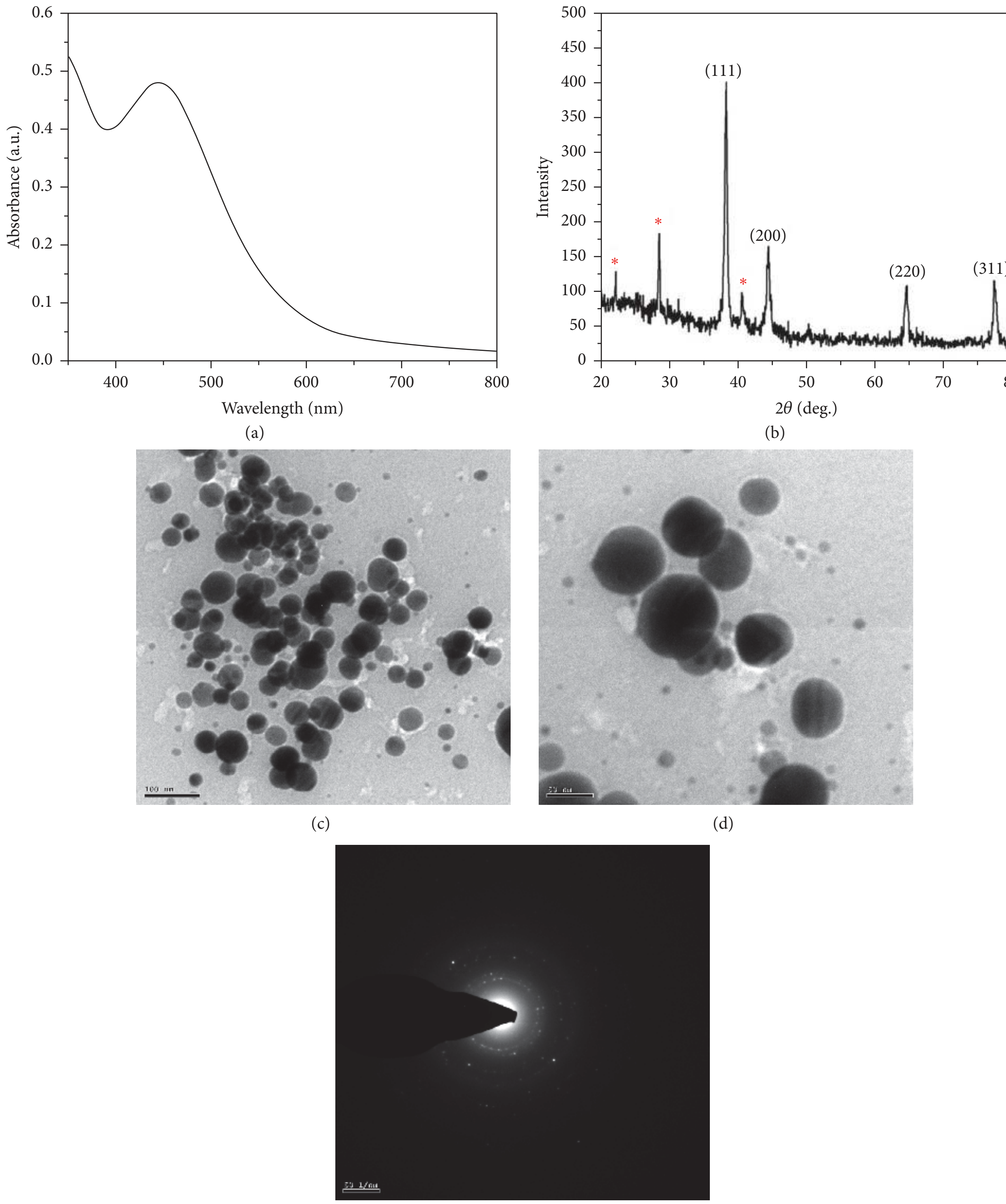

Figure 1: Characterization of silver nanoparticles from *Solanum lycopersicum*. (a) Optical absorption spectra for prepared Ag NPs. (b) X-ray diffraction pattern of prepared Ag NPs. (c) and (d) TEM micrograph of Ag NPs synthesized by using the extract of *S. lycopersicum*. (e) Selected area electron diffraction showing the characteristic crystal planes of elemental silver.

temperature 30°C. The reduction process was performing extremely quickly which altered into reddish brown color for colloid formation. Furthermore, this formation is also due to the excitation of surface plasmon resonance (at 445 nm) effect and therefore the reduction of $AgNO_3$ is complete. Thus, the stable $AgNO_3$ is shaped. Then the characterization of the nanomaterials exhibits unique optical property performed by UV-vis spectroscopy (Figure 1(a)). Moreover,

Table 1: Effect of different concentrations of Ag NPs on adult female *M. rosae*.

Treatment (ppm)	Percentage mortality days after treatment*			
	1st	2nd	3rd	4th
200	10.0 (±5.78)[a]	30.0 (±5.78)[a]	50.0 (±5.77)[a]	80.0 (±5.78)[a]
300	30.0 (±5.80)[be]	50.0 (±5.88)[be]	70.0 (±5.87)[b]	90.0 (±5.80)[ac]
400	40.0 (±5.87)[cef]	60.0 (±5.79)[cef]	90.0 (±5.84)[ce]	100.0 (±5.85)[bc]
500	50.0 (±5.88)[df]	70.0 (±5.87)[df]	100.0 (±5.86)[de]	
Control	0.0	0.0	0.0	0.0

*Mean of 3 replications/treatments; figures in the parentheses show standard error (±SE); means followed by same letters in each column are not significantly different by LSD at 5%.

the XRD pattern of the prepared Ag NPs clearly observes the diffraction peaks at 38.20°, 44.39°, 64.52°, and 77.43° in the 2θ range corresponding to (111), (200), (220), and (311) reflection planes which were exactly indexed to the face centered cubic (fcc) structure of metallic silver, respectively (JCPDS 04-0783) (Figure 1(b)). The observed peaks at 22.11°, 28.43°, and 40.63° in 2θ range indicate the presence of ascorbic acid denoted by the "$*$" (JCPDS 22-1560 and 32-1637) in the *S. lycopersicum* extract. Generally, the broadening of peaks in the XRD patterns of solids is attributed to particle size effects [30]. Broader peaks signify smaller particle size and reflect the effects due to experimental conditions on the nucleation and growth of the crystal nuclei [31]. The average crystallite sizes denote the lattice constant which is calculated by "Bragg's law"; that is, $2d\sin\theta = n$ times wavelength values. For this reason, we have taken XRD pattern of our samples to know d-spacing values of the said nanoparticles. The lattice parameter for the prepared silver nanoparticles works out to be $a = 4.0864$ Å which is in very high-quality concurrence in the standard data file JCPDS number 04-0783.

Therefore, TEM images of the reduced Ag NPs of *S. lycopersicum* are shown in Figures 1(c) and 1(d). It shows the formation of spherical shaped nanoparticles. The strong interaction between number of biomolecules in the tomato fruit extract and surface of nanoparticles was sufficient to the formation of spherical nanoparticles preventing them from sintering and providing size reduction of spherical nanoparticles. The presence of lattice fringes helps to produce the crystalline nature of the prepared nanomaterials. The selected area of electron diffraction pattern of *S. lycopersicum* is shown in the inset of Figure 1(e) which reveals that the rings could be indexed based on the fcc structure of silver which revealed the crystalline nature of the nanoparticles.

Thus, the XRD pattern for Ag NPs denotes the peaks at 38.20°, 44.39°, 64.52°, and 77.43° in the 2θ range corresponding to (111), (200), (220), and (311) reflection planes which were exactly indexed to the face centered cubic (fcc) structure of metallic silver, respectively (JCPDS 04-0783), in the *S. lycopersicum* extract. The sharpening of the peaks clearly indicates that the particles are the spherical nanoparticles (confirmed by the TEM). The average size of the Ag NPs is estimated by using Debye-Scherrer's formula $D = 0.9\lambda/\beta\cos\theta$, where "$\lambda$" represents X-ray wavelength (0.1541 nm), "β" is the maximum intensity and "θ" is Bragg's angle.

Then the bioassay was followed for assessing the insecticidal property of Ag NPs The results showed that 200 and 300 ppm concentrations produced the highest percentage mortality of 80% and 90%, respectively, on the 4th day after treatment (Table 1 and Figure 2). At 400 ppm concentration, mortality reached a maximum of 100% on the 4th day after treatment. However, at 500 ppm concentration, mortality reached a maximum of 100% only on the 3rd day after treatment. The maximum mortalities of 50%, 70%, and 100% were obtained at 500 ppm on the 1st, 2nd, and 3rd day, respectively, after the application of Ag NPs (Table 1). There were statistically significant differences among the treatments on each day after application of biogenic Ag NPs. On 1st, 2nd, and 3rd day 400 and 500 ppm produced significantly higher mortalities than 200 and 300 ppm treatment concentrations (Table 1). On the 4th day, 400 ppm produced significantly higher mortality than 200 ppm and 300 ppm treatment concentrations. There was almost no mortality in the control aphids (Figure 3(a)). Moreover, one-way ANOVA on rose aphid, *M. rosae* experiment with Ag NPs, proposed the following suggestion as mortality on 24, 48, 72, and 96 hrs; F and P values were highly significant. Therefore, it exposes a tremendous effect of the synthesized nanomaterials on rose aphid (Table 1 and Figures 3(a)–3(f)).

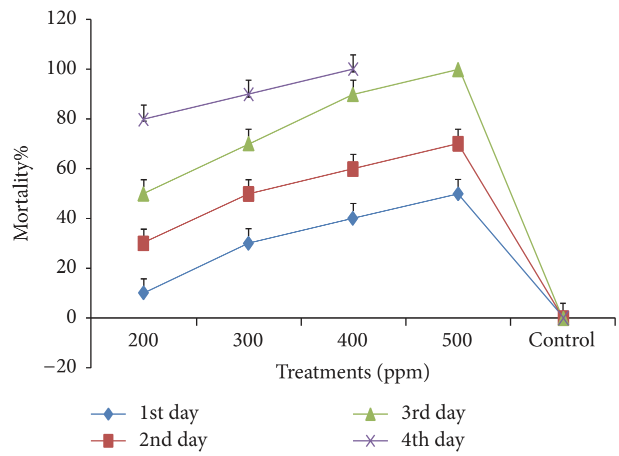

Figure 2: Effect of different concentrations of silver coated nanoparticles on *M. rosae*.

The above results clearly show that all the nanoparticle treatments and mortality of adult aphid increased by increasing the dose of the nanoparticle solution. The Ag NPs should

(a)

(b)

(c)

(d)

(e)

(f)

Figure 3: Insecticidal activity. (a) Aphids in the control experiment. (b) *M. rosae* treated with 500 ppm concentration of Ag NPs. (c) Dead *M. rosae* on the 1st day after treatment of 500 ppm. (d) Dead *M. rosae* on 2nd day after treatment of 500 ppm. (e) Dead *M. rosae* on 3rd day after treatment of 500 ppm. (f) Dead *M. rosae* on 4th day after treatment of 500 ppm.

be tested at concentrations higher than 300 ppm to determine the effective dose resulting in 50% aphid mortality. In terms of time and quantity of Ag NPs required, it is, therefore, acceptable to recommend 500 ppm concentration producing 50% aphid mortality within the shortest possible time with minimal crop damage/injury.

4. Discussions

Our results clearly denote that the Ag NPs of about 13 nm size have the capability to expose the maximum mortality in *M. rosae* (rose aphid). The experimental data also revealed that the 500 ppm has the ability to expose the maximum mortality.

It has been reported that Ag NPs possess insecticidal properties due to changes in its morphological and structural features in relation to surface area [32, 33]. The consequences of the experiment also showed that 200 and 300 ppm concentration of Ag NPs produced the higher percentage mortality of 80% and 90%, respectively, on the 4th day after treatment which indicates the initiation effect of the nanomaterials for exposition of its insecticidal property. At 400 and 500 ppm concentration we can observe the mortality increases. It has been reported that the accumulation of silver can lead to adverse effects on growth, because of their different physicochemical properties and free ions released from Ag NPs [34, 35]. Moreover, the maximum mortalities like 50%, 70%, and 100% were obtained at 500 ppm on 1st, 2nd, and 3rd day of experiment, respectively, after the treatment of Ag NPs. The experiment, however, exposes the dose dependent mortality and there is a possibility that Ag NPs enter into the physiology of rose aphid *M. rosae* which leads to altering the cell physiology and thus leads to mortality. It is very pertinent to consider the reports of Huk et al. [35] that the nanoparticles encompass the ability to enter in the cells which cause cellular damage and cell death [36]. It may be due to necrosis of the said insect cell and thus leads to mortality of the alleged insect pests. Though more experiments are essential to know the actual cause of death of aphids, however, as far as our knowledge is concerned, this is the first report to control the rose aphid by Ag NPs. Therefore, this can be reflected as valuable eco-friendly tools in insect pest management programs, especially on *M. rosae* in Kashmir Valley.

5. Conclusions

We have developed a fast, eco-friendly, and convenient method for biogenic synthesis of Ag NPs from silver nitrate using *Solanum lycopersicum* at room temperature. Color changes from colorless to brownish due to surface plasmon resonance during the reaction with *S. lycopersicum* extract solution resulting in the formation of Ag NPs, which is confirmed by UV-vis spectroscopy. TEM micrograph has shown spherical Ag NPs of size about 13 nm are obtained. Further studies will be conducted to study the pest control management of the synthesized Ag NPs from *S. lycopersicum*.

Competing Interests

The authors declare that they have no conflict of interests.

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