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GREEN SYNTHESIS OF SILVER NANOPARTICLES MEDIATED BY CINNAMON EXTRACT AND ITS POTENTIAL INSECTICIDAL EFFECT AGAINST *CALLOSOBRUCHUS MACULATUS* (F.) (COLEOPTERA: CHRYSOMELIDAE)

Fatma Nur ELMA [1]*, Masood HUSSAIN [2], Ahmet AVCI [3], Erol PEHLİVAN [4], Syed Tufail Hussain SHERAZI [5], Siraj UDDIN [6]

[1] *Selçuk University, Department of Plant Protection, Konya, Türkiye*
[2] *University of Sindh, National Center of Excellence in Analytical Chemistry, Jamshoro, Pakistan*
[3] *KTO Karatay University, Department of Mechatronics Engineering, Konya, Türkiye*
[4] *Konya Technical University, Department of Chemical Engineering, Konya, Türkiye*
[5] *University of Sindh, National Centre of Excellence in Analytical Chemistry, Jamshoro, Pakistan*
[6] *University of Karachi, HEJ Research Institute of Chemistry, International Center for Chemical and Biological Sciences, Karachi, Pakistan*
**** Corresponding Author:*** *fdundar@selcuk.edu.tr*

ABSTRACT

Silver nanoparticles were successfully synthesized using cinnamon extract as a reducing agent. The synthesized nanoparticles, coated with cinnamon extract, were characterized through various optical and spectroscopic techniques. UV-visible spectroscopy confirmed the formation of cinnamon-extract-coated silver nanoparticles (Cinnamon-AgNPs) by optimizing parameters such as precursor salt concentration, pH, temperature and extract volume. The crystalline structure of the nanoparticles was examined using X-ray diffraction (XRD), while size distribution was analyzed through transmission electron microscopy (TEM). It was observed that cinnamon extract effectively stabilized silver nanoparticles and the average particle size was 23.3 nm, with a near-spherical shape. Advances in nanotechnology have recently offered novel approaches in plant protection strategies. The increasing resistance of stored-product pests like *Callosobruchus maculatus* to conventional insecticides necessitates the exploration of eco-friendly alternatives. In this study, the insecticidal activity of silver nanoparticles coated with cinnamon extract was evaluated against the adult stage of *Callosobruchus maculatus*. Additionally, the aqueous extract of cinnamon was also evaluated. Toxicity assays were conducted at varying concentrations of the nanoparticles and cinnamon extract, with exposure durations of 24, 48, and 72 hours. The results revealed that cinnamon-extract-coated silver nanoparticles exhibited the highest toxic effect at the highest concentration after 72 hours (60.72%). In contrast, the aqueous extract of cinnamon did not exhibit a significant toxic effect on *C. maculatus*. This significant difference highlights the synergistic insecticidal effect of the combination of silver nanoparticles and cinnamon extract. Overall, the findings highlight the significant potential of cinnamon-extract-mediated silver nanoparticles as an effective insecticidal agent against *Callosobruchus maculatus*.

Keywords: *Callosobruchus maculatus, Cinnamomum sp., Extract, Silver nanoparticles, Toxicitiy,* XRD.

1 INTRODUCTION

Nanomaterials research is a fascinating and distinctive field of science that plays an essential role in the advancement of modern technologies. The fabrication of metal nanoparticles offers exceptional properties by manipulating their size at the nanometer scale, which distinguishes them from their bulk materials. Metal nanoparticles have found applications in various domains, including agriculture [1], optical devices [2], photonics [3], biolabelling [4], biomedical [5], electrochemical analysis [6], biosensing [7], catalysis [8], information storage [9], and sensors [10]. Numerous synthesis methods have been reported in the literature, including, photochemical reduction [11], solvothermal [12], sonochemical [13], microwave-assisted [14], electrochemical [15], continuous-flow procedures [16], chemical reduction [17], physico-chemical [18], phytosynthesis [19], and thermal decomposition [20]. These traditional methods of metal nanoparticle synthesis often result in environmental contamination due to their reliance on hazardous chemicals and solvents.

In contrast, biosynthetic approaches to produce metal nanoparticles are considered environmentally friendly because they do not rely on harmful substances [21]. Recently, there has been a shift to bio-based nanoparticle synthesis, which is favored for its non-toxic and sustainable properties. Several plant extracts have been employed as both reducing and stabilizing agents in the synthesis of silver nanoparticles, including extracts from *Annona squamosa* peel extract, *Artocarpus heterophyllus*, *Curcuma longa* tuber, *Salvia fruticosa*, *Syzygium cumini*, *Cinnamomum zeylanicum* bark, *Hibiscus rosa-sinensis*, and other extracts. These extracts replace hazardous solvents by utilizing proteins, sugars, and flavonoid-based reducing agents [22], [23], [24].

The cowpea beetle, *Callosobruchus maculatus*, is a cosmopolitan insect pest. Its infestation often begins in the field when the mature pods dry, and once harvested and stored, the pest population rapidly increases, leading to destruction within 3-4 months [25], [26]. While chemical methods have effectively controlled these insects, continuous use has led to numerous negative side effects. As a result, alternative approaches to chemical insecticides have gained attention. Nanotechnology has emerged as a promising modern approach for insect pest management over the past two decades [27]. Recent studies in this field aim to increase pesticide effectiveness, reduce environmental pollution, and promote sustainable agricultural practices. For example, Dura *et al*. [28] reported that combining silver nanoparticles with plant extracts reduces the size of active substances in the plant content, allowing them to pass through the cell walls of harmful microorganisms, thereby enhancing efficacy against pests. Plant-derived nano-formulations have been tested against various insect pests [29], [30], [31], [32], [33], but studies comparing the toxic effects of classical and nano-formulations synthesized from plants remain limited [34], [35], [36], [37]. Thus, the current study is the first to evaluate the toxic effects of silver nanoparticles (Ag NPs) synthesized with *Cinnamomum* extract, as well as the extract alone (prepared with water), against the adult stage of *Callosobruchus maculatus* (Coleoptera: Chrysomelidae).

2 MATERIAL AND METHOD

2.1 Chemicals and Reagents

During this study, high-purity chemicals and reagents were employed. NaOH (98%) and HCl (37%) were procured from Macron Fine Chemicals, while silver nitrate ($AgNO_3$, 97% pure) was sourced from Sigma-Aldrich. All reagents were directly used without any additional purification. Standards and solutions were prepared with deionized water, and all experimental procedures were performed at room temperature.

2.2 Fabrication and Characterization of plant extract protected AgNPs

The synthetic protocol involved the preparation of cinnamon root extract powder; this was achieved by grinding a sufficient quantity of cinnamon using a pestle and mortar. Fifteen grams of the powder were dissolved in 1000 mL of deionized water and left to sit for one day. After filtration, the extract was utilized to synthesize cinnamon-capped silver nanoparticles. For the one-step synthesis, 1.0 mL of 0.1 M $AgNO_3$ was mixed with 6.0 mL of deionized water in a beaker under magnetic stirring for 2 minutes. Subsequently, 3.0 mL of cinnamon root extract was added while stirring vigorously for 5 minutes. The mixture was left undisturbed to stabilize the nanoparticles.

A range of analytical techniques were employed to characterize the biosynthesized silver nanoparticles. An optimization study was conducted using a U-3900 spectrophotometer to refine reaction parameters. The crystalline structure of the extract-protected AgNPs was analyzed via X-ray diffraction (XRD) using a JES-FA/300 instrument. Morphological analysis was conducted using a high-resolution transmission electron microscope (HR-TEM, JEOLS).

2.3 Preparation of cinnamon extract

The preparation of cinnamon extract was conducted according to the method described by Velayutham *et al*. [38]. Fifty grams of powdered cinnamon samples were placed in Erlenmeyer flasks along with 500 mL of deionized water (previously boiled and cooled). The flasks were shaken using an electric shaker for 3 hours. The liquid phase was filtered through Whatman No. 1 filter paper. Deionized water was utilized for all dilutions prepared from the stock solution as well as for the control treatments.

2.4 Insect rearing

Adults of *Callosobruchus maculatus* were taken out from laboratory mass cultures reared in glass jars at 28 ± 2°C and relative humidity of 60±10% on cowpea. Newly emerged adults of *C. maculatus* (1-2 days old) from the laboratory culture were used for the experiments.

2.5 Toxic effect of cinnamon extract embedded AgNPs against Callosobruchus maculatus

During the laboratory trial, cinnamon extract and synthesized AgNPs were subjected to a dose–response bioassay for toxic effects on *Callosobruchus maculatus*. Different concentrations ranging from 5, 10, 20, 40 and 80 mg/l were prepared by diluting in sterile distilled water for synthesized AgNPs. Additionally, the extract of Cinnamon were prepared by diluting in sterile distilled water with 20, 40, 60, 80 and 100 mg/l concentrations. For each experimental treatment, 20 individuals were transferred to each of three Petri dishes. The aqueous cinnamon extract and synthesized AgNPs were topically applied to body surfaces by using a microsyringe as 2 µl for each concentration. Three replications were carried out for each concentration and control. Mortality was recorded every 24 hours for 3 days and the mortality data was corrected by using Abbot's formula [39].

3 RESULTS AND DISCUSSION

3.1 Synthesis and Characterization

UV-vis absorption of silver nanoparticles was confirmed in the spectra at 200-700 nm and analysis was carried out to optimize various reaction parameters. For this purpose, the optimization of concentration of salt was initially carried out and on the basis of more blue-shifted band in UV-vis spectra. Consequently, 0.3ml of 0.1M AgNO3 was optimized for further study as depicted in Fig.1 (a). The observed plasma resonance peaked around at 435 nm, indicating the presence of silver nanoparticles in the solution [40], [41]. The inset photograph revealed that the initial solution was dark, and changes in concentration influenced the nanoparticle coloration. pH was identified as a critical factor in nanoparticle Based on peak shifts, pH 7 was chosen, as shown in Fig. 1(b), with corresponding color changes during the experiment also displayed in the inset.

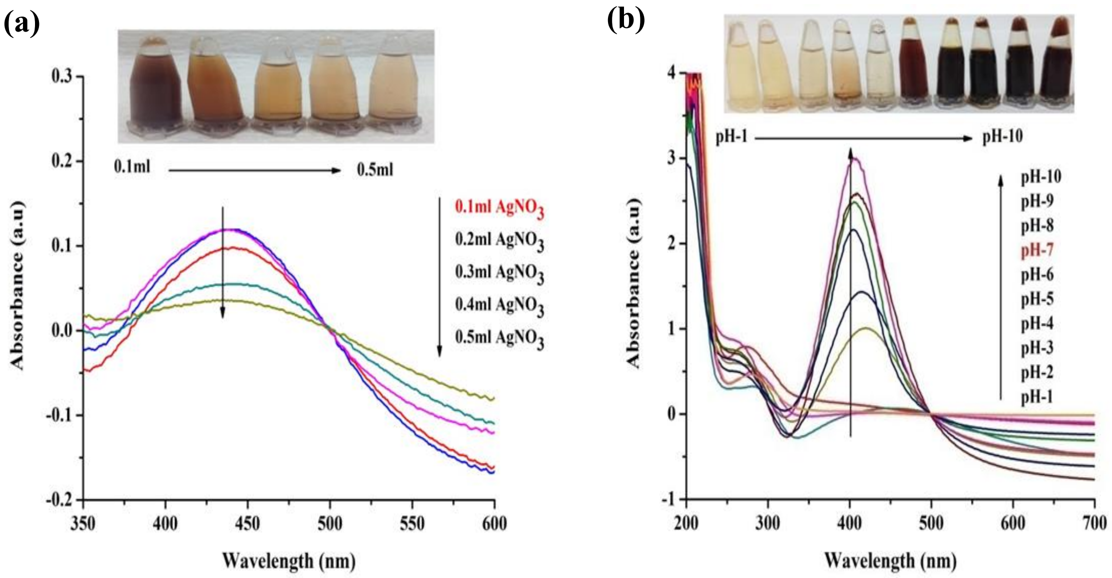

Figure 1. Optimization of 0.1M AgNO3 (a) and effect of pH on formation of cinnamon root extract capped AgNPs (b).

The optimization studies resulted in the production of small, narrowly distributed silver nanoparticles. The crystalline structure of the synthesized nanoparticles was confirmed using XRD analysis, with results presented in Fig. 2. The data indicated that the silver nanoparticles exhibited distinct crystalline peaks at 2Θ values of 38°, 44°, 63°, and 78°, corresponding to the (111), (200), (220), and (311) planes, respectively.

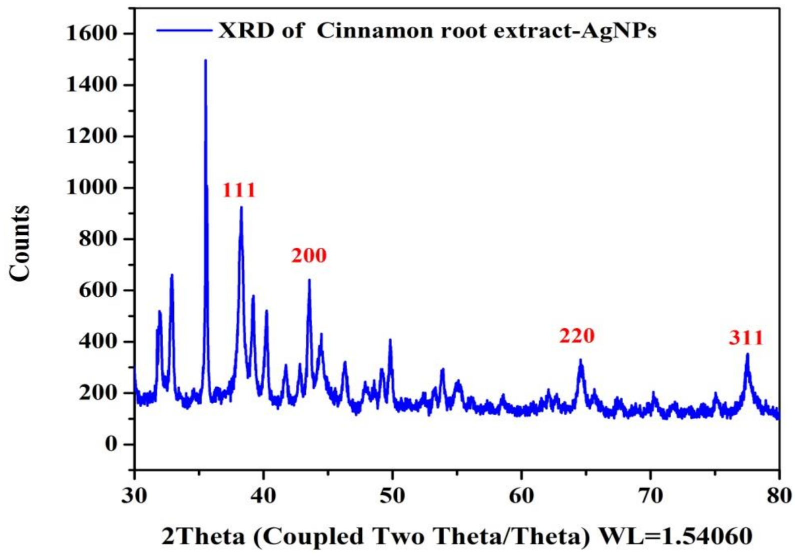

Figure 1. XRD pattern of Cinnamon root extract-protected silver nanoparticles.

Morphological analysis was conducted using a TEM instrument. The findings confirmed that the cinnamon extract effectively stabilized the silver nanoparticles, with an average particle size of 23.3 ± 2 nm, as depicted in Fig. 3(a) and (b). Furthermore, Fig. 3 highlighted the embedding of AgNPs within the cinnamon root extract matrix. The TEM images in Figure 3b show that the cinnamon-based nanoparticles are well-dispersed, round-shaped, and distinct from each other without aggregation.

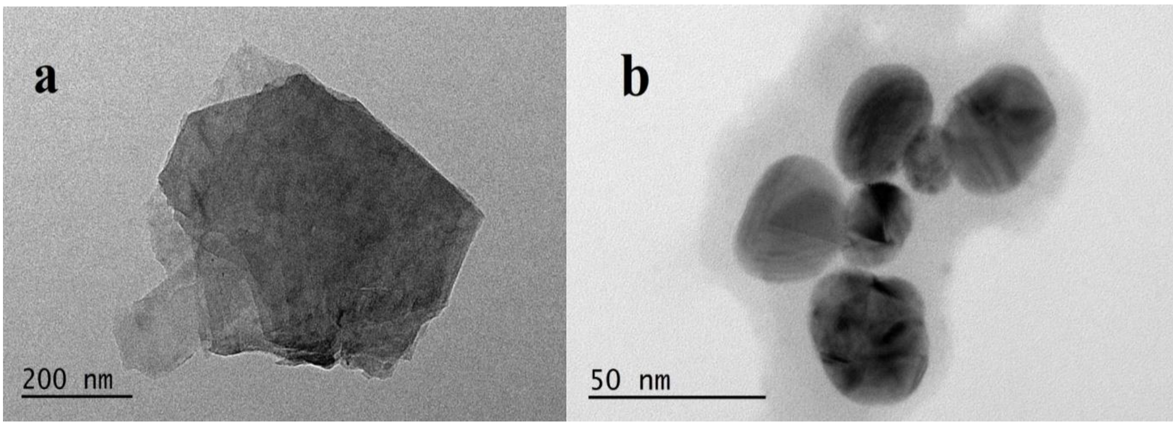

Figure 3. TEM images of a pure cinnamon root extract (a) and cinnamon root extract embedded AgNPs.

3.2 Toxic effects of Cinnamon embedded AgNPs against *Callosobruchus maculatus*

In this study, silver nanoparticles (AgNPs) were successfully synthesized using a green synthesis method with cinnamon extract. The insecticidal effect of the synthesized AgNPs was evaluated, and the results demonstrated that AgNPs coated with cinnamon extract exhibited significant toxicity against *Callosobruchus maculatus*. The synthesized AgNPs showed higher mortality compared to the aqueous cinnamon extract, with a dose-dependent increase in effectiveness. The highest mortality rate (60.72%) was observed after 72 hours at the highest AgNP concentration. In contrast, the aqueous cinnamon extract alone exhibited a lower impact on *C. maculatus*, with mortality rates ranging from 12% to 33.33% at 48 and 72 hours (Table 1).

Table 2. Mortality of Callosobruchus maculatus adult at various concentrations of aqueous extract and synthesized AgNPs using Cinnamon.

Extract	**Concentrations (mg/L)**	**24 h Mortality[a] %±SD**	**48 h Mortality[a] %± SD**	**72 h Mortality[a] % ± SD**
Aqueous extract	20	3.34±1.20	12.06±1.67	14.04±1.02
	40	8.34±1.38	17.23±1.56	17.54±1.61
	60	10±1.24	24.13±1.26	28.07±1.89
	80	10±1.74	28.57±1.45	29.83±1.49
	100	11.66±0.96	30.35±1.58	33.3±1.69
Synthesized AgNPs	5	15±2.01	29.30±1.68	42.85±2.45
	10	23.3±1.98	34.48±2.86	48.21±2.05
	20	30±1.36	41.38±1.96	51.78±2.86
	40	35±2.28	51.72±2.35	57.14±2.78
	80	43.3±1.22	56.90±1.94	60.72±2.13

[a]The mortality was adjusted using Abbott formula

To our knowledge, no previous studies have investigated the insecticidal effects of cinnamon-derived silver nanoparticles (AgNPs) against *Callosobruchus maculatus*. While the toxicity of various nanoparticles towards *C. maculatus* and other insects (e.g., mosquitoes, rice weevils) has been reported [42], [38], [43], [44], [45], [33] research on the comparative insecticidal efficacy of plant-derived nano- and classical formulations remains limited [34],[35], [36], [37]. Balcı et al. [37] demonstrated that nano-formulations of *Melaleuca alternifolia* and *Azadirachta indica* extracts exhibited enhanced efficacy against *Tetranychus urticae* compared to their classical counterparts. Similarly, Al-Husseini et al. [39] reported that silver nanoparticles derived from *Punica granatum* displayed more significant greater toxicity against *Culex quinquefasciatus* than the corresponding ethanol extract. Our findings contribute to this limited body of knowledge by providing evidence of the insecticidal potential of cinnamon coated AgNPs against *C. maculatus*.

4 CONCLUSION AND SUGGESTIONS

A green and economical synthesis method was developed to create stable cinnamon-embedded silver nanoparticles (Ag NPs), using cinnamon as a dual protecting and reducing agent. This approach offers the advantages of simplicity, economic viability, environmental friendliness, ease of use, and high effectiveness. The resulting Ag NPs were characterized using advanced analytical techniques. Analysis revealed that the nanoparticles were uniformly small and highly stable.

Finally, the toxicity of the cinnamon-coated Ag NPs was assessed against adult *Callosobruchus maculatus* (Coleoptera: Chrysomelidae). Toxicity was evaluated over 24, 48, and 72-hour periods, using various concentrations of aqueous cinnamon extract (both with and without Ag NPs). The results demonstrated that the synthesized Ag NPs (at the highest concentration of 80 mg/L) exhibited maximal toxicity after 72 hours (60.72% mortality). In contrast, the cinnamon extract alone showed no significant effect on *C. maculatus*. However, the Ag NPs coated with cinnamon extract demonstrated a significant toxic effect on adult *C. maculatus*.

These findings, showing the significant toxicity of the cinnamon-coated Ag NPs, suggest their potential as botanical insecticides targeting *C. maculatus*. Further research is required to determine the precise mechanism of action and evaluate their efficacy under warehouse storage conditions. Future studies should also assess the effects on non-target organisms and consider the long-term environmental implications.

Conflict of Interest Statement

There is no conflict of interest between the authors.

Statement of Research and Publication Ethics

The study is complied with research and publication ethics.

Artificial Intelligence (AI) Contribution Statement

This manuscript was entirely written, edited, analyzed, and prepared without the assistance of any artificial intelligence (AI) tools. All content, including text, data analysis, and figures, was solely generated by the authors.

Contributions of the Authors

F. N. Elma contributed to the experimental studies, data interpretation, literature review and the preparation of the manuscript.

M. Hussain contributed to the experimental studies and the preparation of the manuscript.

A. Avcı contributed to the experimental studies, the preparation of the manuscript and editing.

E. Pehlivan contributed to the experimental studies.

S. T. H. Sherazi contributed to designing the study.

Sirajuddin contributed to designing the study.

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