RESEARCH PAPER

Evaluation of Pesticide Toxicity in Agricultural Runoff Water: Implications for Aquatic Ecosystem Health

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Abstract

Pesticide residues transported from agricultural fields into aquatic environments can adversely affect fish and other aquatic organisms, leading to physiological stress, altered behaviour, impaired growth and reproduction, and disruption of aquatic ecosystem balance. Malathion, an organophosphate pesticide, is of particular concern because of its widespread agricultural application and potential toxicity to aquatic life. In the present study, green-synthesized nickel oxide nanoparticles were prepared through a plant-mediated synthesis approach and characterized using physicochemical techniques. The synthesized nickel oxide nanoparticles were investigated for their potential to remove malathion from agricultural runoff water under varying nanoparticle dosages. The concentration of residual malathion in the treated water was determined using High-Performance Liquid Chromatography (HPLC). The results showed that nickel oxide nanoparticles exhibited effective adsorption of malathion, with removal efficiency influenced by the nanoparticle dosage. The study demonstrates the potential of green-synthesized nickel oxide nanoparticles as an efficient adsorbent for reducing malathion contamination in agricultural runoff and thereby contributing to the protection of aquatic ecosystems.

Keywords: Plant-mediate synthesis; Nickel oxide nanoparticles; Agricultural runoff; Malathion toxicity; Water treatment.

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Introduction

Agricultural intensification has increased the dependence on pesticides for maintaining crop yield and controlling insect infestations. Following application, a fraction of these chemicals can be transported away from agricultural fields through surface runoff, rainfall, and irrigation drainage and eventually enter nearby streams, ponds, rivers, and other receiving water bodies. The introduction of pesticide residues into aquatic environments is of particular concern because aquatic organisms may be exposed continuously through contaminated water and sediments. Such exposure can interfere with physiological functions, growth, reproduction, behaviour, and survival, thereby affecting the structure and stability of aquatic ecosystems.

Fish are among the organisms particularly vulnerable to pesticide contamination because their continuous contact with the surrounding water facilitates the uptake of dissolved contaminants. Organophosphate pesticides can interfere with the nervous system by inhibiting acetylcholinesterase, an enzyme essential for normal nerve impulse transmission. Consequently, pesticide exposure may produce behavioural abnormalities, neurological stress, reduced feeding activity, impaired growth, and reproductive disturbances in fish [1]. The accumulation of pesticide residues within aquatic food chains can further increase ecological concerns and may indirectly affect organisms at different trophic levels. Therefore, reducing pesticide concentrations in agricultural runoff before their discharge into natural water bodies is important for protecting aquatic biodiversity and maintaining water quality.

Malathion is a widely used organophosphate insecticide employed for the control of a broad range of agricultural and household pests. Although it is considered less persistent than several organochlorine pesticides, its extensive use can result in its occurrence in agricultural drainage and runoff. Once introduced into aquatic systems, malathion may exert toxic effects on fish and other non-target organisms, particularly when exposure occurs repeatedly or at elevated concentrations [3]. Its ability to interfere with acetylcholinesterase activity makes the effective management of malathion-contaminated water an important environmental requirement. Accordingly, the development of efficient methods for removing malathion from agricultural runoff is essential for reducing its transfer to aquatic ecosystems.

Several physicochemical and biological technologies have been explored for pesticide-contaminated water, including membrane separation, biological degradation, coagulation-flocculation, advanced oxidation, and adsorption. However, the suitability of these approaches depends on the nature and concentration of the contaminant as well as the composition of the treated water. Some conventional methods may involve high energy or operational requirements, generation of secondary waste, membrane fouling, or complicated treatment procedures. Adsorption, in comparison, offers a relatively simple and adaptable approach for contaminant removal and can be performed under mild operating conditions [5]. The efficiency of an adsorption process is strongly influenced by the surface characteristics, availability of active sites, and physicochemical properties of the adsorbent.

Nanostructured metal oxides have gained increasing interest as adsorbents because their small particle dimensions provide a high proportion of surface-active sites and allow interactions with a variety of pollutant molecules. Nickel oxide nanoparticles possess distinctive surface and chemical properties that make them attractive for environmental applications. Their adsorption behaviour can be influenced by factors such as surface charge, particle size, surface functional groups, and interaction between the pollutant and nanoparticle surface. These characteristics provide a basis for exploring nickel oxide nanoparticles as potential materials for the removal of organic contaminants from water.

The method employed for nanoparticle preparation is also important when considering their environmental application. Conventional nanoparticle synthesis may involve chemical reducing, stabilizing, or capping agents that can introduce additional chemical residues and increase the environmental burden of the preparation process. Green synthesis has therefore emerged as an alternative approach in which biological materials such as plant extracts provide naturally occurring phytochemicals that can participate in nanoparticle formation and stabilization. Plant-mediated synthesis offers a comparatively simple route for producing nanomaterials while reducing dependence on potentially hazardous synthetic reagents [7]. The incorporation of a green synthesis strategy with an adsorption-based water treatment approach is consequently relevant to the development of more environmentally compatible remediation technologies.

Despite the growing interest in nanomaterial-based pesticide removal, a substantial proportion of adsorption studies has been performed using laboratory-prepared pesticide solutions. Such solutions provide controlled experimental conditions but may not adequately represent the chemical complexity of actual agricultural runoff. Real runoff water can contain dissolved organic substances, inorganic ions, suspended matter, and other agricultural contaminants that may compete for adsorption sites or influence pollutant–adsorbent interactions [8]. Consequently, the performance of an adsorbent established using a single-component laboratory solution may differ when applied to an environmentally derived water sample. Investigations using authentic agricultural runoff are therefore important for establishing the practical relevance of nanomaterial-based adsorption systems.

In this context, the present study investigates the potential of green-synthesized nickel oxide nanoparticles for the adsorption-based removal of malathion from agricultural runoff water. The synthesized nickel oxide nanoparticles were characterized using appropriate physicochemical techniques to establish their morphological, and functional group properties. The adsorption performance was evaluated at different nanoparticle dosages, and the residual malathion concentration was determined using High-Performance Liquid Chromatography.

Materials and Methods

Materials

Nickel sulphate ($NiSO_4$) was used as the nickel precursor for the synthesis of nickel oxide nanoparticles. Fresh ginger rhizomes were collected and used as the reducing agent during nanoparticle synthesis. Sodium hydroxide was used to adjust the pH of the reaction mixture. Agricultural runoff water containing malathion was collected and used for the adsorption experiments.

Preparation of ginger extract

Fresh ginger rhizomes were thoroughly washed with tap water followed by deionized water to remove adhering dust and impurities. The cleaned rhizomes were cut into small pieces and homogenized using a laboratory blender without the addition of water. The resulting slurry was filtered through Whatman No. 1 filter paper to obtain a clear ginger extract. The freshly prepared extract was collected in a clean container and stored at 4 °C until further use in the synthesis of nickel oxide nanoparticles.

Green synthesis of nickel oxide nanoparticles

Nickel sulphate (0.1 M) solution was prepared using deionized water and used as the precursor for nanoparticle synthesis. Subsequently, 20 mL of freshly prepared ginger extract was added to the precursor solution under continuous magnetic stirring. The pH of the reaction mixture was adjusted using 2 M NaOH until the formation of a green precipitate was observed. The resulting suspension was continuously stirred to ensure complete reaction and then allowed to settle. The precipitate was separated by filtration, thoroughly washed several times with deionized water followed by ethanol to remove residual impurities, and dried in a hot-air oven to obtain crystalline green-synthesized nickel oxide nanoparticles.

Characterization of green-synthesized nickel oxide nanoparticles

FTIR spectroscopy was performed to identify the functional groups involved in the green synthesis of nickel oxide nanoparticles and to confirm the formation of metal–oxygen bonds. The FTIR spectrum of the synthesized nanoparticles was recorded over the wavenumber range of 4000–400 cm^{-1} using an FTIR spectrometer. The morphology, particle size, and microstructural characteristics of the synthesized nickel oxide nanoparticles were examined using TEM. A dilute nanoparticle suspension was prepared in ethanol, ultrasonicated to ensure uniform dispersion, and a drop of the suspension was placed onto a carbon-coated copper grid. After drying under ambient conditions, the sample was analyzed using a transmission electron microscope.

Collection of Agricultural Runoff Water

Agricultural runoff water was collected from cultivated agricultural fields in Kanyakumari District, Tamil Nadu. The samples were collected in clean polyethylene containers, transported to the laboratory, and stored at 4 °C until further use. Before the adsorption experiments, the water samples were filtered through Whatman No. 1 filter paper to remove suspended particulate matter. The filtered samples were subsequently used for the adsorption studies. The collected agricultural runoff water was naturally contaminated with pesticide residues, and the presence of malathion was subsequently confirmed by HPLC analysis prior to the adsorption study.

Adsorption of malathion from agricultural runoff water

The adsorption of malathion was evaluated using agricultural runoff water as the test matrix. Aliquots of the water sample (200 mL) were transferred into individual conical flasks, followed by the addition of nickel oxide nanoparticles at concentrations of 5, 10, 20, and 40 mg. The initial pH of the runoff water was recorded as 7.3. Each suspension was maintained under constant agitation at 500 rpm for 120 min at 25 ± 2 °C. After completion of the treatment period, the nanoparticle-containing suspensions were passed through 0.22 μm membrane filters. The resulting filtrates were collected and subjected to HPLC analysis for quantification of malathion. The chromatographic responses obtained before and after treatment were compared to determine the extent of malathion removal at each nickel oxide nanoparticle dosage.

HPLC analysis

Malathion residues in the runoff water were quantified by HPLC using an Agilent 1260 chromatographic system fitted with a UV detector. Prior to injection, the samples obtained after nickel oxide nanoparticle treatment were clarified by passage through a 0.22 μm membrane filter. The filtered aliquots were introduced into the chromatographic system, and detection was performed at 210 nm. The resulting chromatograms were examined for the characteristic malathion peak, and the corresponding chromatographic response was compared with that of the untreated runoff water to determine the change in malathion concentration following adsorption treatment.

Results and Discussion

Characterization of green-synthesized nickel oxide nanoparticles

TEM analysis

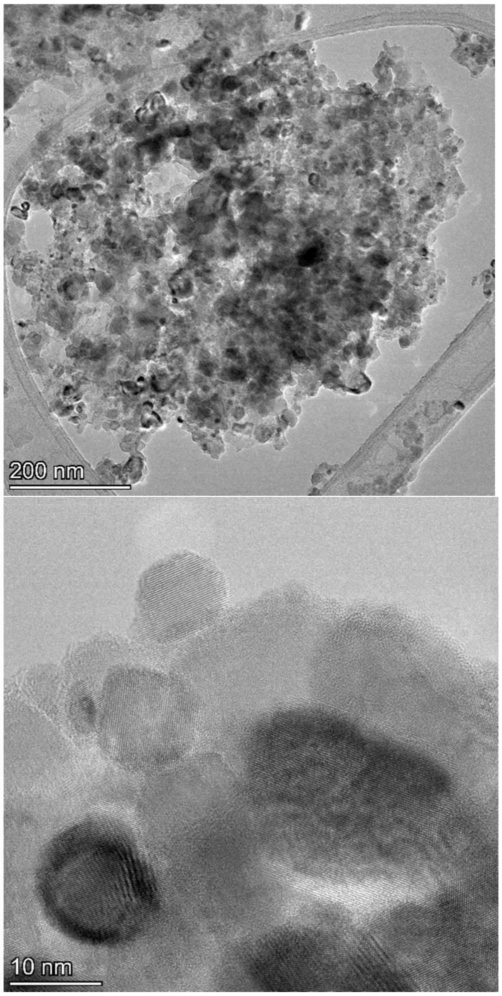


Fig. 1 TEM micrographs of green-synthesized nickel oxide nanoparticles

Transmission electron microscopy (TEM) was used to examine the morphological characteristics and dimensions of the synthesized nickel oxide nanoparticle s. The micrographs showed irregularly distributed nanoparticles with predominantly near-spherical morphology, accompanied by localized particle aggregation. The particle dimensions estimated from the TEM images ranged from 10 to 30 nm, indicating the formation of nanoscale nickel oxide particles. The aggregation observed in some regions may be associated with particle–particle interactions and the high surface energy characteristic of nanomaterials. The higher-magnification micrograph provided clearer visualization of the individual nanoparticles and their boundaries. The nanoscale dimensions observed by TEM indicate the availability of a substantial surface area, which may facilitate interactions between the nickel oxide nanoparticle surface and malathion molecules during the adsorption process.

FTIR Spectroscopy

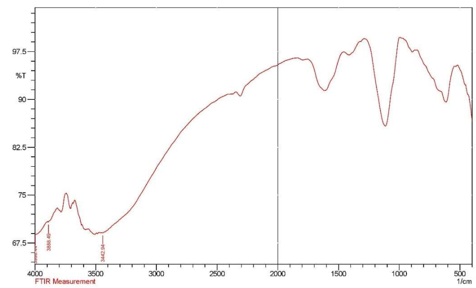


Fig. 2 FTIR spectrum of green-synthesized nickel oxide nanoparticles

The FTIR profile of the synthesized nickel oxide nanoparticles exhibited several absorption features associated with both surface-bound phytoconstituents and the metal oxide framework. The broad band centred at approximately 3423 cm^{-1} can be related to O–H stretching, suggesting the contribution of hydroxyl-containing compounds and adsorbed moisture. An absorption feature around 1620 cm^{-1} may be associated with H–O–H bending and vibrations of oxygen-containing organic moieties originating from the plant extract. The band detected near 1380 cm^{-1} can be assigned to C–H deformation and/or C–N-related vibrations, whereas the feature at approximately 1100 cm^{-1} is attributable to C–O stretching of residual plant-derived compounds. A distinct absorption region between 520 and 550 cm^{-1} corresponds to the characteristic Ni–O vibration, providing spectroscopic evidence for the formation of the nickel oxide phase. The presence of organic functional groups together with the Ni–O band indicates the retention of phytochemical residues on the nanoparticle surface following the plant-mediated synthesis process.

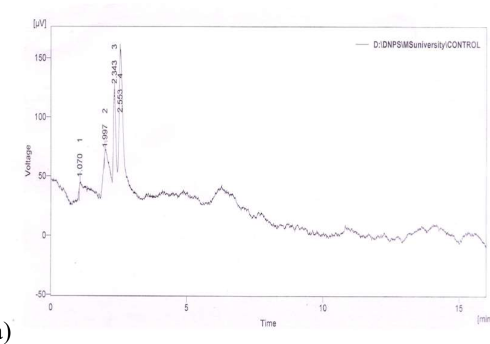
Adsorption mechanism

The removal of malathion from agricultural runoff can be explained by the surface characteristics of the synthesized nickel oxide nanoparticles and the chemical structure of the pesticide. The nanometric particle size provides a considerable surface-to-volume ratio, allowing greater exposure of surface sites to the contaminant present in the water. Functional groups retained from the plant-mediated synthesis may also contribute to the surface characteristics of the nanoparticles and influence their interaction with malathion.

The molecular structure of malathion contains several oxygen- and sulphur-bearing groups that can participate in interactions with the nickel oxide surface. The phosphoryl and ester functionalities may interact with surface nickel centres, while oxygen-containing groups can associate with hydroxyl functionalities present on the nanoparticle surface. Such surface–molecule interactions can promote the attachment of malathion to the adsorbent and contribute to its retention from the aqueous phase. Other weak intermolecular interactions may additionally support the adsorption process. The HPLC results indicated a reduction in the malathion peak intensity following treatment with nickel oxide nanoparticles, with a more pronounced decrease observed at higher nanoparticle dosages. This behaviour indicates greater removal of malathion with increasing adsorbent availability. The chromatographic profile did not show distinct additional peaks attributable to degradation products under the experimental conditions, supporting the interpretation that the observed reduction in malathion concentration primarily resulted from its retention on the nickel oxide nanoparticle surface.

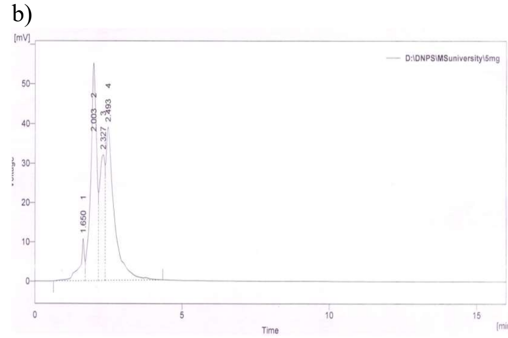
HPLC Analysis of Malathion Adsorption

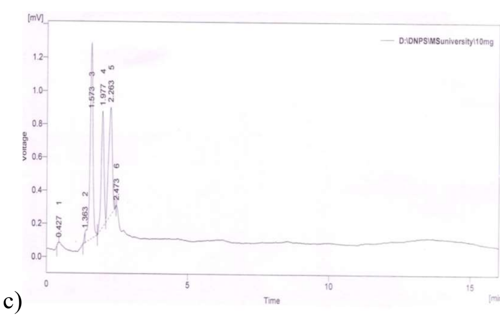
The HPLC chromatograms obtained for the untreated and nickel oxide nanoparticle-treated runoff water showed noticeable differences in the malathion response. A distinct signal was observed in the chromatogram of the untreated sample, establishing the occurrence of malathion in the agricultural runoff. After exposure to the synthesized nickel oxide nanoparticles, the intensity of the corresponding chromatographic signal varied according to the amount of adsorbent introduced into the water sample.



a)

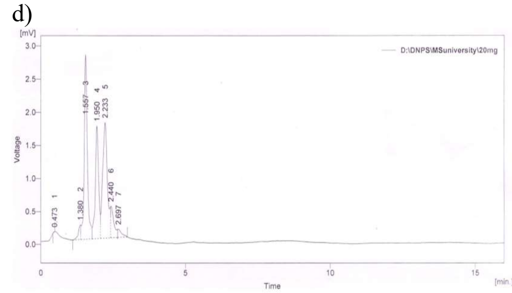
b)





c)

d)



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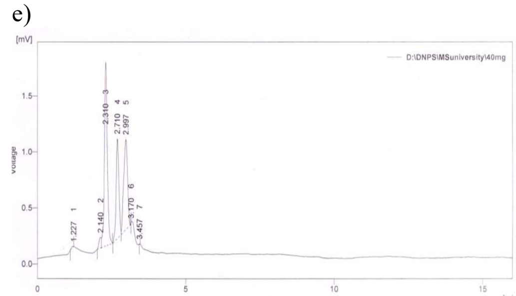

Fig. 3 HPLC chromatograms of untreated agricultural runoff water and samples treated with different dosages (a)5, b)10, c)20 and d)40 mg) of green-synthesized nickel oxide nanoparticles

Among the investigated treatments, the sample containing 40 mg of nickel oxide nanoparticles exhibited the lowest malathion response, whereas comparatively higher responses were observed at lower nanoparticle dosages. The observed trend indicates that increasing the quantity of nickel oxide nanoparticles improved the removal of malathion from the runoff water. This improvement may be associated with the increased availability of nanoparticle surface sites capable of retaining pesticide molecules.

The malathion signal appeared at a comparable retention position in the chromatograms obtained before and after treatment, confirming consistent chromatographic identification of the analyte. The treated samples did not exhibit prominent new signals within the monitored chromatographic profile. Thus, under the conditions employed in the present investigation, the decrease in the malathion response can be interpreted primarily in terms of its removal from the aqueous phase through interaction with the nickel oxide nanoparticle surface.

Conclusion

Green-synthesized nickel oxide nanoparticles prepared using ginger extract exhibited promising potential for the removal of malathion from agricultural runoff water. FTIR and TEM analyses confirmed the formation and nanoscale characteristics of the synthesized material, while HPLC analysis revealed a clear reduction in malathion concentration following treatment, particularly with increasing nickel oxide nanoparticle dosage. The observed removal can be associated with the interaction of malathion molecules with the available surface sites of the nanoparticles. The effective reduction of malathion is environmentally relevant because minimizing pesticide residues in agricultural runoff can reduce their transfer into surrounding water bodies and thereby help limit exposure of fish and other aquatic organisms. Thus, the present findings highlight the potential application of green-synthesized nickel oxide nanoparticles as an adsorbent for reducing pesticide contamination and supporting the protection of aquatic ecosystems.

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