

GREEN SYNTHESIS OF NICKEL NANOPARTICLES USING POMEGRANATE PEEL FOR CR(VI) REMOVAL FROM WASTEWATER: A SUSTAINABLE AND LOW-COST APPROACH

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Abstract

This study successfully synthesized nickel nanoparticles (Ni@NPs) utilizing an aqueous extract of pomegranate (*Punica granatum* L.) peel waste as an environmentally friendly, cost-effective bioreductant and stabilizing agent. The produced nanoparticles were evaluated using UV-Vis spectroscopy, Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Scanning Electron Microscopy with Energy Dispersive X-ray analysis (SEM-EDX). The crystallization of Ni@NPs with nanoscale dimensions was validated using X-ray diffraction and scanning electron microscopy investigations. The efficacy of green-synthesized Ni@NPs in eliminating hexavalent chromium Cr(VI) from wastewater was assessed by time-dependent batch tests. Under optimal conditions, the nanoparticles attained a maximum Cr(VI) removal effectiveness of 95.2% in 150 min. Kinetic analysis demonstrated that the removal process adhered more closely to a pseudo-first-order model ($R^2 = 0.9783$, $k_1 = 0.0198 \text{ min}^{-1}$), suggesting a mechanism dominated by chemisorption. This study illustrates a dual sustainability benefit: the valorization of agro-waste from fruit processing and the synthesis of an efficient, environmentally benign nanomaterial for heavy metal remediation, thereby advancing green chemistry and circular economy principles.

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Introduction. The hexavalent form of chromium, Cr(VI) is one of the most dangerous contaminants due to its remarkable toxicity and environmental persistence. Cr(VI) is a common and hazardous pollutant in industrial effluents, soils, and drinking water sources worldwide due to the extensive use of chromium compounds in industries like electroplating, leather tanning, pigment production, metallurgy, and wood preservation, which has resulted in massive discharges of wastewater containing chromium over decades [1, 2]. The hexavalent form of chromium, which persists in the environment for long periods and poses significant risks to both humans and wildlife, is carcinogenic, genotoxic, and mutagenic in living organisms, making exposure to Cr(VI) extremely harmful to human health. The World Health Organization (WHO) has set a maximum content limit of 0.05 mg L⁻¹ for Cr(VI) in drinking water due to its significant toxicity [3].

Traditional remediation approaches for Cr(VI), such as chemical precipitation, ion exchange, coagulation, membrane filtration, and reverse osmosis, have significant limitations [4]. These physicochemical approaches are costly, produce secondary chemical sludge, and are efficient solely at elevated heavy metal concentrations, hence constraining their use in practical wastewater treatment contexts. These constraints have heightened the pursuit of sustainable, efficient, and economically viable alternatives. Nanotechnology has arisen as a revolutionary framework to tackle this issue [5].

Green synthesis of nanoparticles has garnered a lot of attention among the various synthesis methods as an inexpensive, ecologically friendly, and sustainable alternative to conventional physical and chemical processes [6–8]. Plant extracts, bacteria, fungi, algae, and other biological agents are used to provide natural reducing and stabilizing biomolecules for the synthesis of metal and metal oxide nanoparticles such as Ag, Au, Cu, and ZnO. These nanoparticles offer exceptional antibacterial, antifungal, anticancer, and catalytic properties due to their distinct physicochemical characteristics and high surface-to-volume ratio [9–11].

Punica granatum L. (Pomegranate) peel is one of the main agro-industrial by-products produced during juice and food processing, making up around 26–30% of the fruit weight [12]. Pomegranate peel has become a great renewable resource for the environmentally friendly production of metal nanoparticles because of its high level of polyphenols, flavonoids, tannins, and other bioactive phytochemicals [13, 14]. The production of environmentally friendly nanomaterials with potential uses in environmental remediation, catalysis, antimicrobial treatments, antioxidant systems, and biomedical fields is made possible by the valorization of this plentiful agricultural waste through nanoparticle synthesis, which also reduces pollution in the environment and encourages sustainable waste management.

Metallic Ni@NPs are one of the many transition-metal nanomaterials used

for Cr(VI) remediation, and they have several unique benefits [15–17]. Compared to non-magnetic adsorbents like activated carbon or charcoal, Ni@NPs have a major practical advantage due to their high saturation magnetization, which enables simple recovery from aqueous media via an external magnetic field. Additionally large number of active sites for both adsorption and reduction pathways are provided by their highly specific surface area and adjustable surface chemistry (oxide layer, hydroxyl groups). Over the past ten years, there has been a significant increase in the green synthesis of Ni@NPs using plant extracts [15]. However, only a small number of studies have been published that focus specifically on the removal of Cr(VI) [17]. To close this gap, the present work focused on the green synthesis of Ni@NPs using pomegranate peel extract as a bio reducing agent, followed by comprehensive physicochemical characterization (XRD, FTIR, SEM, UV-Vis) and evaluation of their efficiency in removing Cr(VI) from wastewater. The findings of this work are expected to contribute to the development of sustainable, low-cost, and scalable nanomaterials for heavy metal remediation.

Materials and methods. Chemicals and reagents. A 0.1 M solution of nickel(II) nitrate hexahydrate, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, was used for the synthesis of the nanoparticles. Potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) was used to prepare Cr(VI) stock solutions. All chemicals were of analytical grade. Deionized water was used in all experimental procedures.

Preparation of pomegranate peel extract. Fresh pomegranate peels (*Punica granatum* L.) were collected from local markets in Tokat, Turkey. They were first washed with deionized water to remove surface impurities and dried at 60 °C for 24 h. The dried plant material was ground into a fine powder. Ten grams of peel powder were added to 100 mL of deionized water and heated to 80 °C for 60 min with continuous stirring. The resulting aqueous extract was cooled and filtered through Whatman No. 1 filter paper, then stored at 4 °C until use.

Green synthesis of nickel nanoparticles. For the synthesis of Ni@NPs, 50 mL of pomegranate peel extract was added dropwise to 100 mL of 0.1 M $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution under continuous magnetic stirring at 80 °C. The mixture was stirred for 2 h until a colour change was observed, indicating nanoparticle formation. The resulting precipitate was centrifuged at 8000 rpm for 15 min, washed three times with deionized water and ethanol to remove residual organic compounds, and dried at 100 °C. The dried powder was calcined at 400 °C for 2 h.

Characterization of Ni@NPs. The optical properties and formation of Ni@NPs were initially confirmed by UV-Vis spectroscopy (200–800 nm). FTIR spectroscopy ($400\text{--}4000\text{ cm}^{-1}$) was employed to identify the functional groups present on the nanoparticle surface and to confirm the role of phytochemicals in capping and stabilization. The crystalline structure and average crystallite size were determined by XRD analysis using $\text{CuK}\alpha$ radiation ($\lambda = 0.15406\text{ nm}$) over a 2θ range of 20 °C–80 °C, with crystallite size calculated using the Scherrer

equation. Surface morphology and elemental composition were analyzed by SEM-EDX.

Cr(VI) removal experiments. A standard Cr(VI) stock solution of 100 ppm (100 mg L^{-1}) was prepared by dissolving 0.283 g of $\text{K}_2\text{Cr}_2\text{O}_7$ in 100 mL of distilled water using a calibrated volumetric flask. The working solutions ($10\text{--}50 \text{ mg L}^{-1}$) were prepared by diluting the stock solution with distilled water. Experiments for removing Cr(VI) were performed as batch studies. Batch studies were performed in open air with continuous stirring at 400 rpm with a mechanical stirrer. A specified quantity of Ni@NPs (2 mg) was introduced to 50 mL of 30 ppm Cr(VI) solution in a conical flask while maintaining continuous agitation at ambient temperature and at the optimum pH of 2 [17]. At $\text{pH} \leq 2$, $[\text{HCrO}_4]$ and $[\text{Cr}_2\text{O}_7]^{2-}$ are the main Cr(VI) ions, with $[\text{HCrO}_4]^-$ having the strongest adsorptive capacity due to its low free energy. The negative charge on the adsorbent surface reduces electrostatic chromium anion adsorption at high pH values. With potassium dichromate salt and better adsorption at low pH, $[\text{Cr}_2\text{O}_7]^{2-}$ will be the medium's chromium ion. Samples were extracted at specified time intervals (0, 30, 60, 90, 120, and 150 min) and subjected to filtration using $0.45 \text{ }\mu\text{m}$ membrane filters. The quantity of Cr(VI) was determined spectrophotometrically at 353 nm using a calibration curve ($(10\text{--}50 \text{ mg L}^{-1})$; $y = 0.125x + 0.333$, $R^2 = 0.9859$). The removal efficiency was evaluated based on absorbance measurements and the corresponding removal percentages.

The removal efficiency (%) was calculated by employing the following formula:

$$\text{Removal (\%)} = [(A_0 - A_t) / A_0] \times 100,$$

where A_0 and A_t are the absorbance values at time zero and time t , respectively.

Results and discussion. Green and biological nanomaterials exhibit superior efficacy in pollution removal due to their low toxicity, extensive surface area, and varied functional groups, while the use of biological agents in their synthesis minimizes environmental impact and amplifies purification potential [18]. Numerous studies have documented the effective use of biogenic metal oxide nanoparticles in the photocatalytic degradation and adsorption-based removal of heavy metal ions from wastewater; however, the targeted application of green-synthesized Ni nanoparticles for the removal of Cr(VI) has yet to be extensively studied in the literature [19].

The surface morphology of the nickel nanoparticles was analyzed using scanning electron microscopy (SEM), which is the elemental composition of the nanoparticles, exhibiting predominant signals for nickel and oxygen, hence affirming the identification and purity of the produced material (Fig. 1). The EDX analysis of Ni@NPs reveals the elemental composition. Ni is the dominant element, constituting 96.5% by atomic percentage, indicating successful formation of Ni@NPs. The trace amounts of Cl were attributed to residual organic substances from the pomegranate peel extract or from the water.

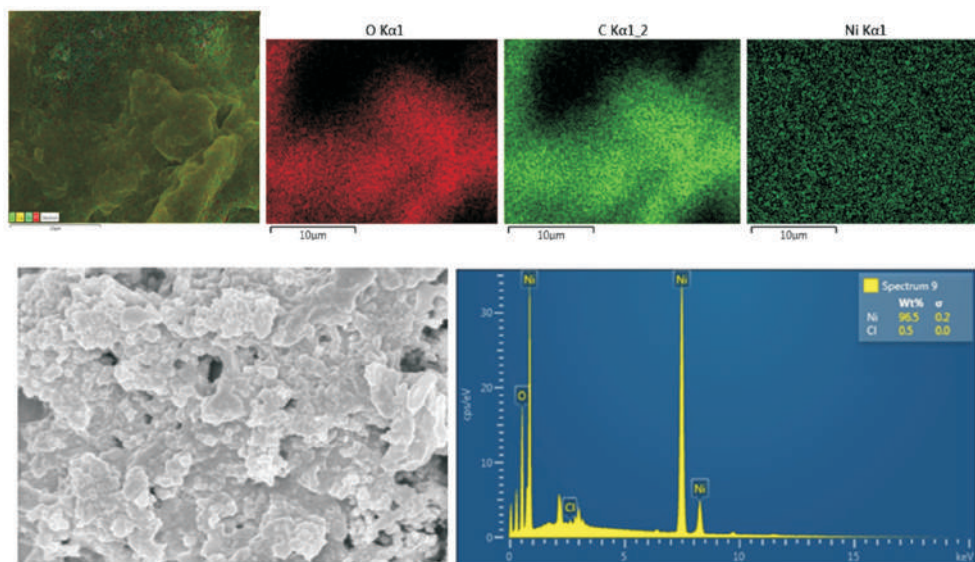


Fig. 1. Elemental mapping, SEM images, and EDX pattern of Ni@NPs

The red map indicates a substantial distribution of oxygen over the sample. Nevertheless, it seems to be more concentrated in some high-relief regions. This indicates the existence of oxides, presumably nickel oxides, or oxygen-containing functional groups on the surface. The green map signifies a prevalent and extensively dispersed carbon signal (from plant materials). The Ni signal is disseminated over the scanned region, indicating that the Ni@NPs or layers are effectively incorporated into the carbon matrix. The distribution seems quite uniform.

X-ray diffraction (XRD) analysis was used to examine the crystalline structure of the biosynthesized Ni@NPs (Fig. 2). The diffraction pattern showed clear

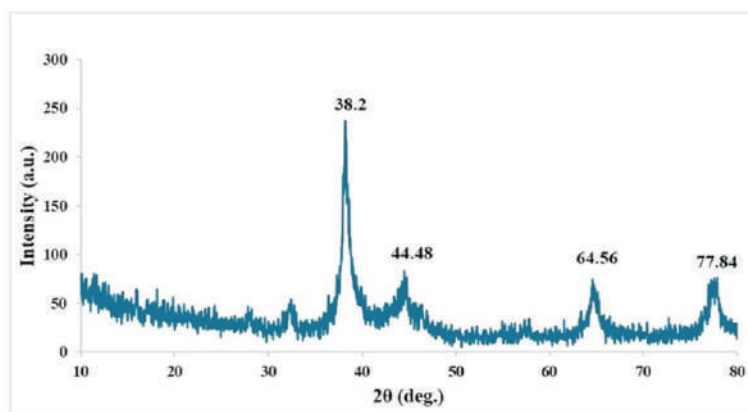


Fig. 2. XRD pattern of the synthesized Ni@NPs

peaks at around $2\theta \approx 38.2^\circ$, 44.48° , 64.56° , and 77.84° , which correspond to the (111), (200), (220), and (311) planes of face-centred cubic (FCC) nickel, respectively. The (111) crystallographic plane of face-centred cubic (FCC) metallic nickel is represented by the strong diffraction peak seen at 44.48° , which is in good agreement with the standard JCPDS Card No. 04-0850. The creation of crystalline Ni nanoparticles is further confirmed by the diffraction peak at 77.84° , which can be indexed to the (311) plane of FCC nickel. Results are comparable with the reported earlier literature [17]. The strong crystallinity and preferred orientation of the produced nanoparticles are demonstrated by the prominent and sharp diffraction peak at about 38° , which corresponds to the (111) plane. Phase-pure nickel nanoparticles were successfully synthesized using the green synthesis approach, as indicated by the appearance of distinct peaks devoid of extra impurity signals. A little widening was seen in addition to the primary diffraction peaks, which is usually linked to nanoscale crystallite size. Lattice strain and the crystallites' limited size, two traits of nanostructured materials, are the causes of this broadening. The Scherrer equation was used to calculate the Ni@NPs' average crystallite size:

$$D = K\lambda/\beta \cos \theta,$$

where θ is the Bragg angle, β is the full width at half maximum (FWHM), K is the form factor (0.9), D is the crystallite size, and λ is the X-ray wavelength (0.15406 nm). The crystallite size was estimated to be around 10–12 nm using the strongest diffraction peak at $2\theta = 38^\circ$. This number is comparable with previously reported, green-synthesized Ni@NPs and verifies that the synthesized particles are within the nanoscale range [17]. High surface area, which is beneficial for catalytic and adsorption applications, especially in heavy metal removal processes, is indicated by the comparatively small crystallite size.

Cr(VI) removal experiments. The remediation studies were done with solar light. Cr(VI) removal experiments were conducted as batch studies. A known amount (2 mg) of Ni@NPs was added to 50 mL of Cr(VI) solution in a conical flask under continuous stirring at room temperature. Samples were withdrawn at defined time intervals (0, 30, 60, 90, 120, and 150 min) and filtered through 0.45 μm membrane filters. Cr(VI) concentration was measured spectrophotometrically at $\lambda = 353$ nm. The degradation percentage and absorbance values were recorded to monitor the removal efficiency over time (Fig. 3).

The mixture was stirred for up to 150 minutes, with aliquots of the supernatant periodically collected for examination. The Cr(VI) uptake mechanism is generally characterized by the pseudo-first-order kinetic model. The rate constants (k) were derived from the slopes of their corresponding linear plots. The computed rate constants and associated regression coefficients (R^2) are summarized in Fig. 3.

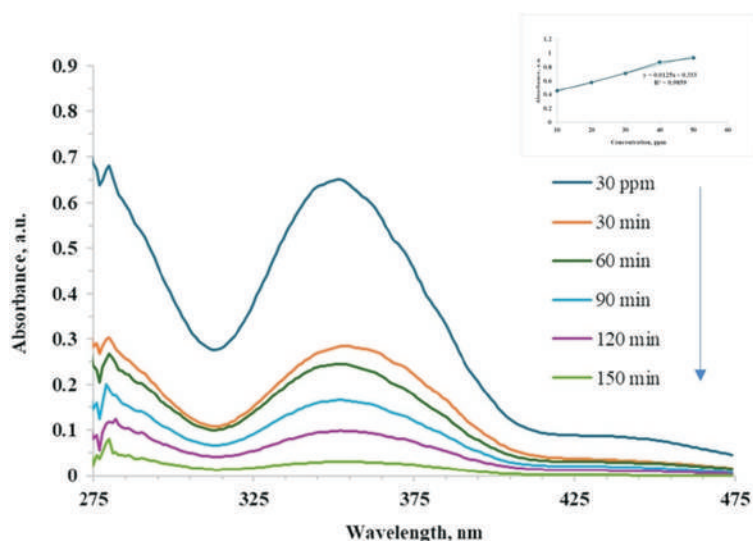


Fig. 3. Results of Cr(VI) adsorption experiments

The collected experimental results at different contact times were utilized to fit pseudo first order model presented in Fig. 4.

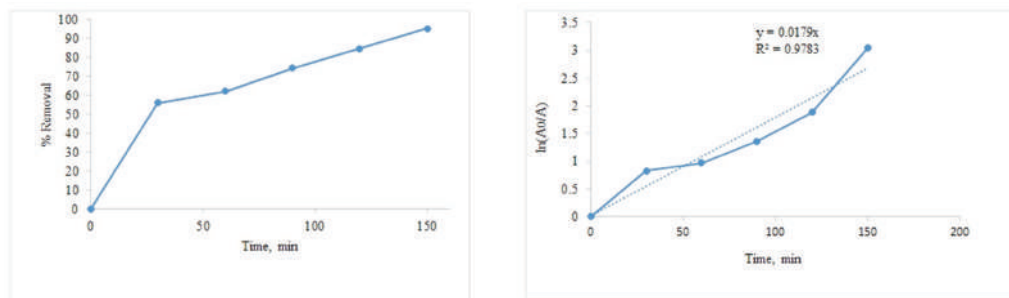


Fig. 4. The adsorption kinetic test for Cr(VI) removal, models of kinetics percentage of removal pseudo first order on Ni@NPs

A pseudo-first-order kinetic model was used to assess the removal kinetics of Cr(VI) by the produced Ni@NPs. The model can be written as:

$$\ln \left(\frac{A_0}{A} \right) = kt,$$

where A_0 and A are the initial and time-dependent absorbance values, respectively, k is the rate constant (min^{-1}), and t is the contact time (min). The experimental absorbance data were converted into $\ln(A_0/A)$ values and plotted against time. The slope of the linear regression plot of $\ln(A_0/A)$ with time was used to get the rate constant (k). Rate constant (k): about 0.0179 min^{-1} and Coefficient of

correlation (R^2): about 0.9783. The high R^2 value indicates excellent correlation between the experimental data and the pseudo-first-order kinetic model.

The degradation efficiency of Cr(VI) increased significantly with time, reaching approximately 95.2% within 150 min, demonstrating the high removal efficiency of the synthesized Ni@NPs.

Conclusion. Despite the proven effectiveness of Ni@NPs in removing contaminants in a number of processes, there is little research into their specific role in the removal of Cr(VI). Ni@NPs attained markedly higher removal efficiency at merely 150 min compared to those documented in the literature. The relatively high-rate constant indicates rapid interaction between Cr(VI) and the active surface sites of the Ni@NPs. This study illustrates a dual sustainability benefit: the valorization of agro-waste from fruit processing and the creation of an efficient, environmentally friendly nanomaterial for heavy metal remediation, which advances green chemistry and circular economy concepts. The promise of the produced Ni@NPs as a useful material for wastewater treatment applications is highlighted by their high removal efficiency, advantageous kinetics, and nanoscale characteristics.

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