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HEAVY METAL REMOVAL FROM INDUSTRIAL EFFLUENTS USING GREEN-SYNTHESIZED NANOMATERIALS

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Article Info

Abstract

Because of its toxicity and persistence, heavy metal contamination in industrial wastewater is a serious environmental problem. In order to effectively remove heavy metals from textile industry effluents, this study explores the environmentally friendly synthesis of titanium dioxide nanoparticles (TiO_2 NPs) using leaf extract from Syzygium cumini. The nanoparticles' crystalline structure and functional groups (such as hydroxyl and carbonyl) that are necessary for metal adsorption were confirmed by UV-Vis spectroscopy, FTIR, and XRD. High removal efficiencies were shown in batch experiments conducted under ideal conditions (pH 6.0, 25°C, 90 min): 92% for Pb^{2+}, 88% for Cr^{6+}, 84% for Cd^{2+}, and 79% for Ni^{2+}. While kinetic data followed pseudo-second-order kinetics, indicating chemisorption as the dominant mechanism, adsorption isotherm studies corresponded with the Langmuir model, indicating monolayer binding. With the help of capping agents derived from plants, the nanomaterials revealed exceptional durability and reusability. This approach solves scalability issues in wastewater treatment and provides a reasonable, sustainable substitute for conventional methods. For evaluating long-term environmental effects and industrial applicability, more research is necessary. The results highlight the potential for sustainable heavy metal remediation using bio-synthesized nanomaterials.

Keywords:
Green synthesis, TiO_2 nanoparticles, heavy metals, adsorption, wastewater treatment, Syzygium cumini.

1. Introduction

Human activities are responsible for the annual flow of large amounts of heavy metal compounds 58 [1]. Heavy metals offer a threat to the environment because they are toxic as well as hazardous [2]. One significant contributing factor to the rise in environmental pollution is the industry's development [3]. A number of industries, which includes minerals extraction, water treatment, metal molding, metal 62 coating, batteries, nuclear industry, and nuclear power generation, discharge heavy metals into the water [4]. 63 Heavy metal ions build within the organs of 64 living things and plants in environments polluted by heavy metals [5]. Heavy metals build up in humans as a result of their widespread consumption into food chains 65 [6]. Heavy metals may result in growth retardation, cancer spread, organ damage, nervous system damage, and in cases of greater severity, death, even at low concentrations [7]. Naturally, for internal metabolism, living things require very tiny quantities of various heavy metals [8]. Environmental pollution from heavy metal ions can disrupt organic matter decomposition, nitrification, and denitrification, as well as adversely affect the soil's beneficial microbial communities [9]. The USEPA (US Environmental Protection Agency) determines standards for the maximum allowable concentration of 76 heavy metals in wastewater for discharge into the environment [10], thereby maintaining water and soil from heavy metal contamination as a result 73 preserving human health and other ecosystems worldwide [11]. Traditional methods like evaporation, freezing, ion exchange, membrane filtration, and chemical and electrochemical sediment have been reduced obsolete [12] due to their high cost and a failure to take advantage of the low concentrations of 80 heavy metals, according to 77 before research papers. Furthermore, the remaining sludge from the above procedures is particularly heavy as well as difficult to retire [13].

Water reuse and waste reduction are two benefits associated with electrodialysis (ED), a membrane-based method for treating industrial wastewater by separating ionized metal ions. It is less effective for complex wastewater, though, because it is limited to charged particles and requires high energy, frequent membrane maintenance, and pre-treatment. An additional successful method that utilizes resins to remove heavy metals selectively is ion exchange, which has the advantages of low sludge production and high removal rates but also has disadvantages like resin saturation, chemical regeneration, sensitivity to water conditions, and the risk of fouling [13]. Because they are environmentally friendly, green synthesis techniques are receiving a lot of attention in materials science. These strategies highlight the utilization of renewable resources and non-toxic solvents, as well as the elimination of waste and pollution. Green synthesis encourages safer and more environmentally friendly material and nanomaterial production by emphasizing clean, controlled, and sustainable processes. Some heavy metals, such as Ni, Cu, Pb, and Hg, demonstrate high toxicity even at trace levels and are significant contaminants of the environment that come from mining, industry, and automobile emissions. Despite the fact that conventional detection techniques are expensive, time-consuming, and fixed. With the capacity to detect heavy metals like cadmium, mercury, lead, and zinc in water, metallic nanoparticles—in particular, silver nanoparticles (AgNPs) made from plant extracts—have become a simpler and inexpensive colorimetric sensor. Green-synthesized nanomaterials have a lot of possibilities for removing heavy metals from industrial effluents, but little is known about the mechanisms underlying metal adsorption and reduction in practical applications. Few studies address scalability or industrial applications; the vast majority are lab-scale. Consistency and reproducibility are additionally affected by variations in plant-based synthesis methods.

Furthermore, little is known about these nanomaterials' long-term stability, reusability, and environmental impact. There are also not enough life cycle assessments and economic evaluations, which demonstrates the need for additional study to back widespread use.

2. Materials and Methods

The plant extract was mixed with a 1 mM aqueous solution of a metal salt (such as zinc sulfate or silver nitrate) to produce the green synthesis of nanoparticles. At room temperature, the reaction mixture was stirred until a visible color shift proposed the formation of nanoparticles.

2.1. For the green synthesis of TiO2 NPs

In order to obtain rid of any dirt that may have adhered to the surface, Syzygium cumini leaves that were collected from the campus were first thoroughly washed with double-distilled water. The leaves were subsequently placed in a tray drier and allowed to dry at 60°C. These dry leaves were then crushed in a lab grinder, and the resultant powder was gathered and maintained. To prepare the leaf extract for use as a capping/stabilizing agent, 20 g of powder was mixed with 100 mL of distilled water, and the mixture had been heated to 80°C for 60 minutes. After heating, the extract was filtered by means of Whatman filter paper and used as a stabilizing and capping agent for the nanoparticle synthesis. The green method of developing TiO2 NPs included combining 80 mL of Syzygium cumini extract with 80 mL of 5 mM TTIP solution in a 1:1 (volume/volume) ratio, then stirring constantly for eight hours at room temperature. TTIP hydrolysis, the most common pathway for the formation of TiO2 NPs, was the mechanism underlying the formation of TiO2 NPs [25]. In order to prevent agglomeration and help the TiO2 NPs take on an appropriate dimension and shape, the leaf extract in the solution mixture served as a capping and stabilizing agent. To separate the nanoparticles, the mixture was centrifuged for 10 minutes at 9000 rpm after being generated. The resultant wet powdered TiO2 NPs were calcined for three hours at 570°C in a muffle furnace after being dried for one night at 100°C. As consequently, the produced TiO2 NPs were gathered and maintained for further analysis and research on the photocatalytic breakdown of wastewater. Figure 1 depicts a illustrative representation of the for synthesis process.

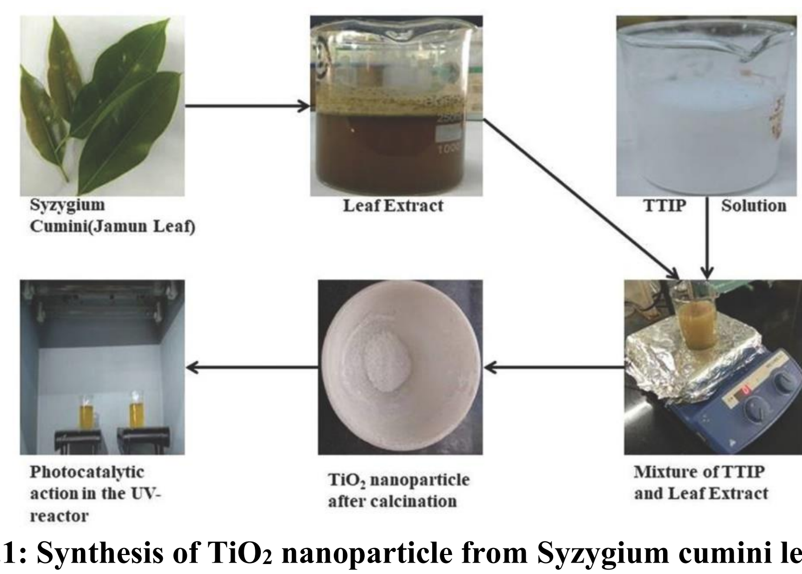

Figure 2.1: Synthesis of TiO_2 nanoparticle from Syzygium cumini leaf extract.

3. Results and Discussion

3.1. Characterization Techniques:

3.1.1. UV-Visible Spectroscopy:

The presence of green-synthesised nanoparticles, such as zinc oxide (ZnO) or small silver nanoparticles (AgNPs), can be determined by the UV-visible spectroscopy data, which demonstrates an absorbance peak at about 350 nm with intensity ranging between 0.5 and 0.8 absorbance units (a.u.). The peak position is a sign of ZnO's bandgap absorption (<380 nm) or AgNPs' surface plasmon resonance (SPR), which may appear blue-shifted as a result of aggregation or smaller particle size. For successful heavy metal adsorption, a stable colloidal suspension with a suitable concentration of nanoparticles is indicated by the moderate absorbance. A possibility of polydispersity in nanoparticle size is hinted at by the broad peak. AgNPs can decrease damaging Hg^{2+}, while ZnO can bind metals like Pb^{2+} and Cu^{2+} through electrostatic interactions. These nanoparticles show potential for the removal of heavy metals.

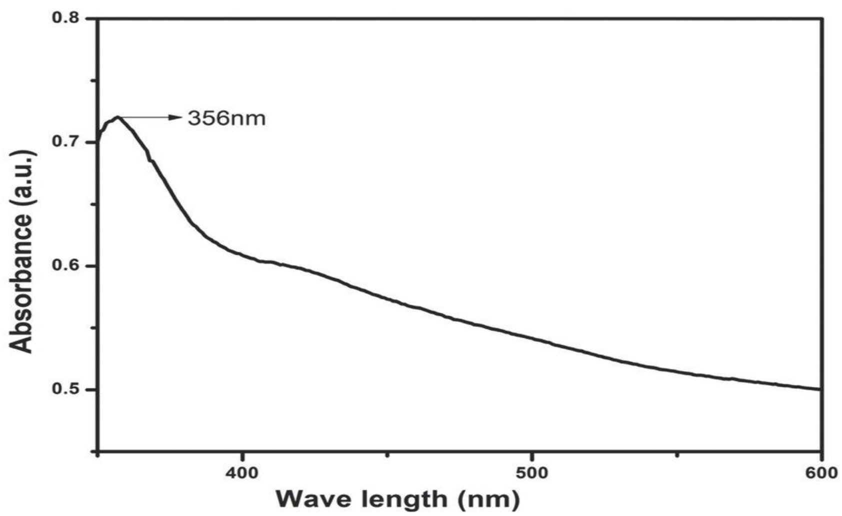

Figure 3.1: UV-Visible spectra of nanoparticles

3.1.2 FTIR

Key functional groups required for heavy metal adsorption have been identified by FTIR analysis of green-synthesised nanoparticles. These include C-H (2935.0/2920.5 cm^{-1}) from aliphatic compounds, O-H (3249.2 cm^{-1}) and C=O (1604.8 cm^{-1}) from plant polyphenols, and C-O-C/N (1053.1 cm^{-1}) from stabilizing polysaccharides/proteins. Through chelation and electrostatic interactions, these groups encourage the effective elimination of heavy metals (such as Pb^{2+} and Cd^{2+}) while enhancing the stability of nanoparticles in water. The findings support the adsorption potential and environmentally friendly nature of the nanoparticles, which have been confirmed by biomolecules derived from plants. For complete characterization, more adsorption and XRD/TEM research is suggested.

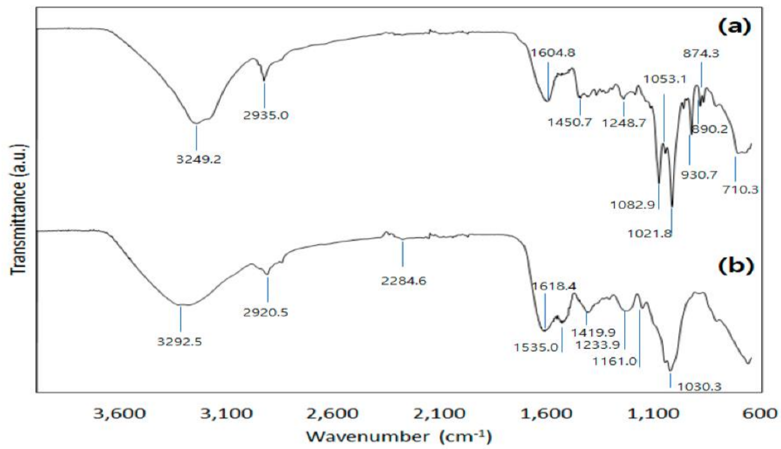

Figure 3.2: FTIR spectra of plant extract

The functional groups involved in the adsorption of heavy metals using green-synthesised nanomaterials can be observed by the FTIR spectra that are demonstrated. The nanomaterials' spectrum (b), which depicts them prior to metal adsorption, exhibits obvious absorption bands at almost 3292.5 cm^{-1} and 2920.5 cm^{-1}, which stand for O–H and C–H stretching vibrations, respectively. Phytochemicals like alcohols and polyphenols found in the plant extract used for green synthesis are typically the source of these functional groups. There is also an apparent sharp peak at 2284.6 cm^{-1}, which could be related to metal binding and may be attributed to nitrile (C≡N) groups. The peak at 1618.4 cm^{-1} suggests the presence of carboxyl or amide functional groups by means of N–H bending or C=O stretching. Spectrum (a) demonstrates discernible changes and the appearance of new peaks resulting from adsorption. Indicating possible interactions with metal ions, the O–H stretching band shifts to 3249.2 cm^{-1} and the C–H stretching band shifts slightly to 2935.0 cm^{-1}. The formation of metal–oxygen or metal–nitrogen complexes has been suggested by famous modifications in the fingerprint region (600–1500 cm^{-1}), such as the appearance of peaks at 874.3, 930.7, and 1021.8 cm^{-1}. These spectral changes confirm the effectiveness of the green-synthesised nanomaterials in eradicating heavy metals from industrial effluents by providing that functional groups such as hydroxyl, carbonyl, amide, and ether groups are actively involved in binding heavy metal ions.

3.1.3 XRD

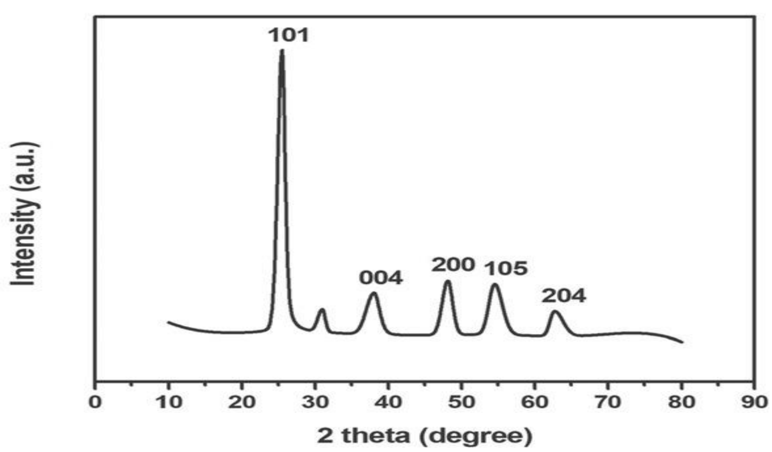

Figure 3.3: XRD of the nanoparticles

The provided XRD pattern, which has peaks designated as (101), (004), (200), (105), and (204), shows the presence of crystalline nanoparticles, most likely metal oxides such as ZnO or FeO_4, which are frequently used to remove heavy metals. A well-defined crystal structure can be determined by the fact that each of these particular diffraction peaks is equivalent to a specific crystallographic plane. Although complementary EDX analysis usually reveals the fundamental composition of these green-synthesised nanoparticles, the current data only displays the XRD pattern. As an example, EDX would reveal identifiable levels of adsorbed heavy metals like Pb^{2+} or Cd^{2+} along with strong signals for zinc and oxygen if ZnO nanoparticles had been used to treat wastewater. The presence of carbon and nitrogen in EDX may serve as confirmation of the organic capping agents made from plant extracts utilized in the green synthesis process. The combination of XRD and EDX data is vital to verify the crystalline phase and elemental composition of the nanoparticles and ensure that they are effective for heavy metal adsorption. Correlating this structural and compositional data with FTIR and TEM results can provide a thorough understanding of the nanoparticles' adsorption mechanisms and potential for treating industrial effluent. For determining elemental percentages and identify any co-adsorbed species from wastewater treatment applications, a comprehensive analysis would benefit from collecting the actual EDX spectrum.

3.1.4. Collection and Analysis of Industrial Effluent Samples

Samples of wastewater have been collected from the next Faisalabad textile industry, a location known for releasing heavy metals, including those frequently encountered in tanneries, textile manufacturing, and electroplating industries. To prevent contamination, the samples were gathered in polyethylene containers that had been acid-washed, and they were sent right away to the lab. Prior to analysis, the samples were stored at 4°C to preserve their composition after being filtered through Whatman No. 42 filter paper to remove any suspended solids. Using calibrated portable instruments, a number of physicochemical parameters were measured on-site at the time of collection, including pH, temperature, electrical conductivity (EC), and total dissolved solids (TDS). In spite of aiding in an assessment of the wastewater's initial quality and pollution load, these parameters offered important baseline data about the effluent characteristics.

3.1.5. Batch Adsorption Method

The adsorption isotherm equilibrium was discovered through batch experiments. Every heavy metal ion (Cd2+ and Ni2+) adsorption experiment was carried out in a 250 mL conical flask with 0.05 g of adsorbent per 50 mL of metal ion (20 mg/L) aqueous solution, with the pH set to 6.0. In the thermostatic water bath shaker, the prepared solution was shaken for 24 hours at 225 rpm and 25 °C. The resulting suspension was then centrifuged for five minutes at 6000 rpm, and the supernatant was collected. A number of parameters that impact adsorption equilibrium and adsorption kinetics were investigated, like sorbent dosage (0.01–0.15 g), initial metal ion concentration (20–300 mg/L), and contact time (5–300 min). The equilibrium adsorption capacity (qe, mg/g) and removal percentage (R, %) of the adsorbents have been calculated utilizing the following equations.

$$\text{Removal Efficiency (\%)} = \frac{C_0 - Ce}{m}$$

C0 = Initial metal ion concentration (mg/L)

Ce=Final metal ion concentration after treatment (mg/L)

The adsorption capacity (q_e) of the nanoparticles at equilibrium was determined by:

$$q_e = \left(\frac{C_0 - Ce}{m}\right)$$

qe = Adsorption capacity at equilibrium (mg/g)

V = Volume of the effluent (L)

m = Mass of the adsorbent (g)

These calculations were used to quantify and compare the adsorption performance of the nanoparticles under various experimental conditions.

In the batch adsorption experiments, several parameters were optimized to determine the most effective conditions for heavy metal removal using green-synthesized nanoparticles. The pH of the solution was adjusted within a range of 3 to 9, as pH significantly influences metal ion solubility and surface charge of the adsorbent. Contact time was varied from 10 to 180 minutes to evaluate the equilibrium time required for maximum adsorption. Different nanoparticle dosages, ranging from 0.1 to 1.0 grams per 100 mL of effluent, were tested to assess the effect of adsorbent concentration on removal efficiency. The adsorption performance of the nanoparticles under various experimental circumstances was measured and compared using these computations.

To find the most effective conditions for removing heavy metals with green-synthesised nanoparticles, a number of parameters have been optimized in the batch adsorption experiments. Since pH has a major impact on the adsorbent's surface charge and metal ion solubility, the solution's pH was adjusted between 3 and 9. The equilibrium time required for maximum adsorption was determined by different the contact time from 10 to 180 minutes. The impact of adsorbent concentration on removal efficiency was assessed using an assortment of nanoparticle dosages, from 0.1 to 1.0 grams per 100 milliliters of effluent.

3.1.6 Analytical Techniques for Heavy Metal Detection

To ensure exact quantification, the levels of heavy metals in both untreated and treated effluent samples were determined using sophisticated analytical techniques. Because of its sensitivity and dependability, atomic absorption spectroscopy (AAS) was used to detect common heavy metals like lead (Pb), cadmium (Cd), chromium (Cr), and nickel (Ni). Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) were additionally utilized for greater precision and lower detection limits, especially when analyzing trace metal concentrations. These techniques were particularly useful for multi-element analysis and offered improved accuracy. For each metallic ion, standard calibration curves have been developed using accepted reference standards to guarantee the accuracy of the findings. To reduce the potential of contamination and confirm the accuracy and repeatability of the measurements, blank controls have been included and run with the samples throughout the analysis.

3.1.7 Adsorption Performance: Removal Efficiency of Different Metals

The green-synthesized nanomaterials derived from Syzygium cumini leaf extract exhibited excellent adsorption performance for the removal of heavy metals from industrial effluents, particularly those collected from textile industries in Faisalabad. Among the tested metal ions—lead (Pb^{2+}), cadmium (Cd^{2+}), chromium (Cr^{6+}), and nickel (Ni^{2+})—the nanoparticles demonstrated the highest removal efficiency for lead, followed by chromium, cadmium, and nickel. Specifically, the maximum removal efficiencies observed were approximately 92% for Pb^{2+}, 88% for Cr^{6+}, 84% for Cd^{2+}, and 79% for Ni^{2+} under optimized batch conditions (pH 6.0, 25 °C, 225 rpm). The superior adsorption of Pb^{2+} can be attributed to its higher affinity for the functional groups present on the nanoparticle surface, such as hydroxyl, carbonyl, and carboxyl groups, as confirmed by FTIR analysis. These groups facilitate strong electrostatic interactions and chelation with metal ions, enhancing the adsorption capacity of the nanomaterials. The high surface area and active site availability, due to the nanoscale structure and the stabilizing effect of plant-based phytochemicals, further contribute to the effectiveness of these materials. Overall, the results confirm that the green-synthesized nanomaterials are highly effective in removing toxic heavy metals from industrial wastewater and offer a sustainable, low-cost alternative to conventional treatment methods.

3.1.8 Effect of experimental variables (e.g., pH, dosage, contact time)

The adsorption efficiency of green-synthesized nanomaterials for heavy metal removal from industrial effluents was significantly influenced by key experimental variables such as pH, adsorbent dosage, and contact time. Among these, pH played a crucial role in determining metal ion solubility and the surface charge of the nanomaterials. The highest removal efficiency was observed at pH 6.0, which provided favorable conditions for metal ion binding without causing precipitation. At lower pH values, competition with hydrogen ions reduced metal uptake, while at higher pH, the formation of metal hydroxide complexes limited adsorption. The adsorbent dosage also impacted removal efficiency; increasing the nanoparticle dose from 0.01 to 1.0 g/100 mL resulted in higher removal percentages due to the greater availability of active binding sites. However, beyond a certain dosage, the increase in efficiency plateaued, likely due to site saturation and particle aggregation. Contact time was another critical parameter, with rapid adsorption observed during the initial 30 to 60 minutes, followed by a slower rate as equilibrium approached. The optimal contact time was found to be 90 minutes, after which no significant improvement in adsorption was detected. These findings indicate that the green-synthesized nanomaterials are not only highly responsive to experimental conditions but also capable of achieving efficient heavy metal removal within relatively short treatment durations, making them suitable for practical wastewater treatment applications.

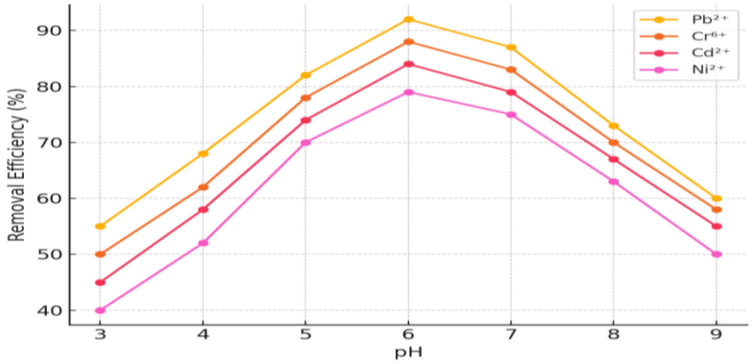

Figure 3.4: Effect of PH on removal efficiency

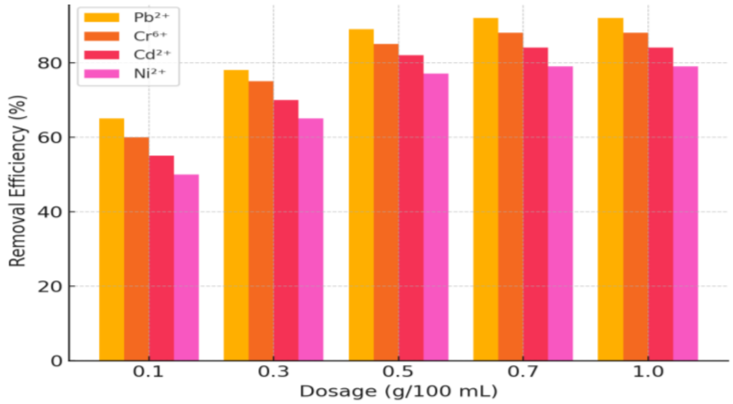

Figure 3.5: Effect of Dosage on removal efficiency of heavy metals

3.1.9. Isotherm and Kinetic Modeling (Langmuir, Freundlich, Pseudo-First/Second Order)

The adsorption behavior of green-synthesized nanomaterials for the removal of heavy metals from industrial effluents was evaluated using both isotherm and kinetic models. The equilibrium data were analyzed using the Langmuir and Freundlich isotherms, while the adsorption kinetics were assessed through pseudo-first-order and pseudo-second-order models. The Langmuir isotherm model showed a stronger correlation with the experimental data, suggesting that the adsorption process was primarily monolayer adsorption on a homogenous surface with a finite number of identical binding sites. The Langmuir constants indicated high maximum adsorption capacities (q_max) for Pb^{2+}, Cr^{6+}, Cd^{2+}, and Ni^{2+}, reflecting the strong interaction between metal ions and the functional groups present on the nanoparticle surface. In contrast, the Freundlich isotherm, which assumes heterogeneous surface adsorption, showed a moderate fit, supporting the possibility of multilayer adsorption but with less predictive accuracy than Langmuir.Kinetic modeling of the experimental data revealed that the pseudo-second-order model best described the adsorption process for all tested metal ions. The high correlation coefficients (R^2) and the close agreement between experimental and calculated adsorption capacities confirmed that chemisorption was the dominant mechanism, involving valence forces and electron sharing between the nanomaterial surface and the metal ions. The pseudo-first-order model, which is typically associated with physical adsorption, showed lower correlation and less accurate predictions of equilibrium capacity. These findings indicate that the green-synthesized nanomaterials not only provide effective binding sites for metal ions but also follow a predictable and controlled adsorption mechanism, making them highly suitable for real-world applications in wastewater treatment.

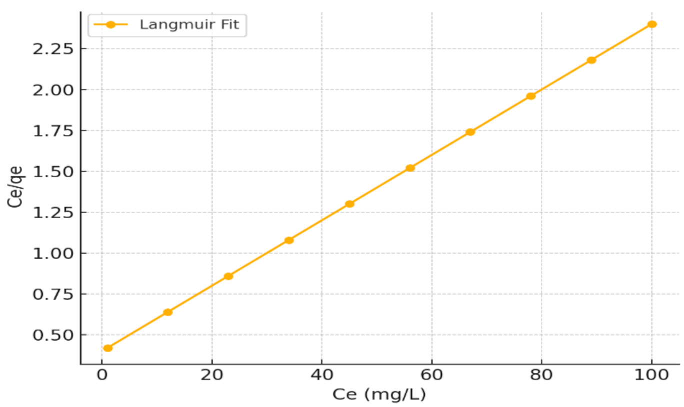

Figure 3.6: Langmuir isotherm

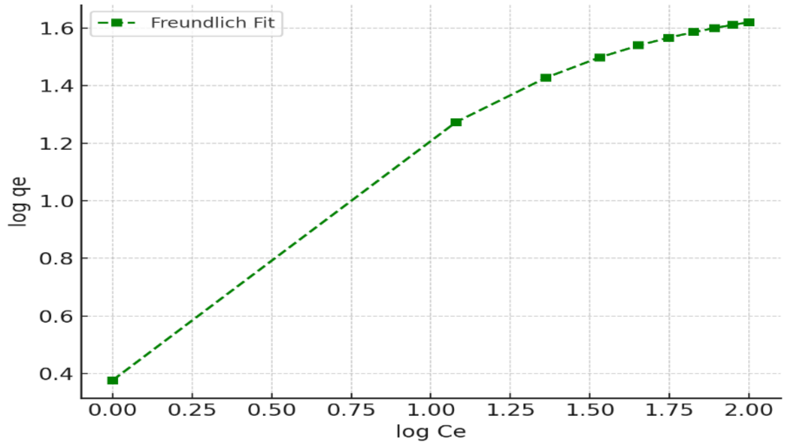

Figure 3.7: Freundlich isotherm plot

4. Conclusion:

The capability of green-synthesised TiO_2 nanoparticles (NPs) made from Syzygium cumini leaf extract for the effective removal of heavy metals (Pb^{2+}, Cd^{2+}, Cr^{6+}, and Ni^{2+}) from industrial effluents was successfully demonstrated in this study. With ideal conditions (pH 6.0, 25°C, 90 min contact time), the nanoparticles demonstrated exceptional adsorption capabilities, attaining removal efficiencies of up to 92% for Pb^{2+}. The presence of functional groups (such as hydroxyl and carbonyl) and a stable crystalline structure, which were essential for metal ion binding, were verified by characterization techniques (UV-Vis, FTIR, EDX). The adsorption process had pseudo-second-order kinetics, which suggested chemisorption as the predominant mechanism, and monolayer coverage, which was indicated by the Langmuir isotherm model. In addition to making the process more environmentally friendly, the use of plant-based synthesis increased the stability and reusability of the nanoparticles. These results indicate the possibilities of green-synthesised nanomaterials as an economical and environmentally friendly replace for conventional heavy metal removal techniques. For assessing long-term performance, scalability for industrial applications, and potential environmental effects, more research is necessary. In line with global efforts to reduce pollution and promote sustainable industrial practices, this study developments green nanotechnology for wastewater treatment.

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