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NI(II) IONS SEQUESTRATION FROM WATER USING PRISTINE AND FUNCTIONALIZED *Daucus Carota*

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ABSTRACT

Nickel (Ni) contamination in water poses serious human health risks, necessitating the development of low-cost, sustainable remediation methods. The main purpose of the current study is to explore naturally occurring materials that can be effectively employed to remediate Ni(II) from water under specific conditions, and it aligns with SDG 6 targets. In this study, carrot (*Daucus Carota*) seed (DCS) and its modified form (MDCS) have been used for the first time as adsorbents for the sequestration of Ni(II) ions from a water sample. Fine seed powder of *Daucus Carota* was modified using 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrochloride (EDC) and L-cystine to enhance the adsorption efficiency. DCS and its modified form were systematically characterised through different tools. FTIR analysis confirms the modification of the seed biomass with L-cystine through the presence of additional functional groups, particularly sulfur-containing moieties, which play a decisive role in enhancing nickel ion adsorption. XRD and SEM studies further confirmed the changes in the pristine material after modification. The adsorption behaviour of DCS and MDCS was analysed through adsorption experiments. Equilibrium data were fitted to adsorption isotherms (Langmuir/Freundlich/Temkin). Adsorption capacity (Langmuir) was found to be 161.29 mg/g and 232.55 mg/g for DCS and MDCS, respectively. Results confirmed that both materials can be used for the removal of Ni(II) ions from water. Furthermore, the green materials, DCS and MDCS, proposed in the current work can be tested for other contaminants and will contribute to the availability of clean drinking water, supporting global efforts toward sustainable water management and environmental protection.

Keywords: Ni(II), Sustainable, L-Cystine, Adsorption, Toxic Metal, Water Treatment, Environment.
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INTRODUCTION

The presence of heavy metal ions in water is of great concern globally due to their adverse effects on human and aquatic health. Metals like chromium, lead, arsenic, nickel, etc., enter the environment through many routes, including mining activities, metal industries, and natural sources.[1] Presence of Ni(II) in water is also a matter of concern due to its high ionic mobility and bioaccumulation in living organisms.[2-4] Ni exposure produces deleterious effects in plants and promotes reactive oxygen species, which leads to oxidative damage.[5-6] The toxic effects of Ni ions are also reported in aquatic ecosystems, leading to ecological risks to aquatic food chains.[7] It has been reported that nickel affects more than 60 human/microbial proteins.[8-10] Due to the toxicity and presence of Ni(II) ions in water in developing countries, effective and low-cost remediation strategies are required. Methods such as chemical precipitation (CP)/membrane filtration (MF)/ion exchange (IE)/electrochemical (EC) may be used for water treatment, but they have high operational costs and energy requirements. In the above context, the adsorption method is among the most efficient and convenient approaches for water treatment, owing to its simplicity, high efficiency, low energy requirements, and low cost. Recent studies increasingly support the use of materials derived from natural and waste biomass as adsorbents due to their abundance, renewability, biodegradability, and rich surface chemistry (Table-1).[11-12] The sorbent capacity of different used materials is listed in Table-1. Biosorbents derived from agricultural fragments, microbial biomass, and biopolymers, including cellulose, chitosan, lignin, and alginate, have also demonstrated promising performance for water treatment.[13-14] The presence of active functional groups, including hydroxyl /carboxyl /amino/ phenolic groups, enables effective metal binding via electrostatic attraction/ion exchange or through surface complexation. Chemical modification to enhance the adsorption properties of natural adsorbents has also been widely explored in various studies, and modification by sulfur-containing compounds has proven effective for metal remediation from water. [24-26] There is a need to

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explore more low-cost green materials with high adsorption efficiency for the remediation of Ni(II) ions at pH 6-8 and room temperature.

Table-1: Sorbent Capacity of Reported Adsorbents

Adsorbent	Sorbent capacity (mg/g)	References
Activated carbon from palm waste	166.7	[14]
Modified activated carbon	210	[15]
Ion-imprinted polymer	117.53	[16]
MgO-modified pomegranate peel biochar	45.5	[17]
Foamed metakaolin-based geopolymer	28.34	[18]
Non-foamed metakaolin-based geopolymer	38.46	[18]
Pomegranate peels	11.4	[19]
Green walnut husk	99	[19]
Eichornia crassipes (water lily) treated with water	349	[20]
Eichornia crassipes (water lily) treated with NaOH	293.8	[20]
Natural Moroccan illite-smectite clay	0.83	[21]
Natural Moroccan illite-smectite clay	0.830	[21]
Walnut shell	13.92	[22]
Carob Seeds	85.73	[23]

Carrot seeds have not yet been investigated for their adsorbent removal efficiency. They are rich in lignocellulosic components, including cellulose/hemicellulose/lignin/ proteins.[27] In this study, carrot - *Daucus Carota* seeds in pristine and functionalized form have been used for sequestration of Ni(II) ions from water. The study proposed the use of sustainable materials for Ni(II) remediation from water and supports Sustainable Development Goal (SDG) 6. The research aims to make a substantive contribution to the field of water treatment by offering practical solutions for the sustainable removal of Ni(II) ions from aqueous solutions.

EXPERIMENTAL

Materials and Methods

Analytical-grade chemicals were used in the current study, purchased from BR Scientific & Chemicals, Faridabad. Nickel(II) nitrate hexahydrate ($Ni(NO_3)_26H_2O$) was used to prepare a bivalent nickel(II) stock solution. H_2SO_4, EDC, and L-cystine ($HSNH_2CH_2CO_2H$) were used for surface modification of *Daucus Carota seed coat*. Carrot seeds (*Daucus Carota*) were collected from the Faridabad local market and used as the adsorbent material.

Preparation of Pristine Adsorbent Material from the *Daucus Carota* Seed Coat

The carrot seeds were thoroughly washed and air-dried at room temperature for 48–50 hours. The seed coat was separated from the dried seeds, and the fine powder was prepared. This carrot-seed–derived powder was used to remove nickel metal ions.

Preparation of Modified *Daucus Carota* Seed

The prepared powder of *Daucus Carota* seed was then modified with EDC and L-cystine to increase the adsorbent's surface activity. In 1.0 g of *Daucus Carota* seed powder, 30 mL of methanol and 30 mL of double-distilled water were added at pH 5, adjusted with sulphuric acid. After that, 100 mg EDC dissolved in 6 mL water, and 2 mL of EDC solution was added dropwise over a period of 30 minutes to the *Daucus Carota* seed powder round-bottom flask. The reaction was stirred at room temperature for an additional 10 minutes. A total of 1 g of L-cysteine hydrochloride was dissolved in distilled water (5 mL), maintaining a pH of 5. L-cysteine hydrochloride solution was added slowly to the *Daucus Carota* and EDC solution. The pH of the final solution was adjusted to approximately pH 5.5. This mixture was refluxed using a reflux condenser with stirring for 6 hours at 65–70 °C on a magnetic stirrer. After 6 hours, the flask was removed, cooled, and centrifuged. The centrifuged residue was washed 6 times with double-distilled water and methanol (1:1) after filtration. The materials after washing were used as modified *Daucus Carota* seed powder (Fig.-1, Scheme-I).

Characterization Studies

Functional group characterisation of *Daucus Carota* seed before and after catalyst modification was done using Perkin-Elmer FTIR. Samples were prepared following the universal attenuated total reflectance (ATR) method, and spectra were recorded (4000–400 cm^{-1}) using a KBr disk as the reference. A scanning Electron Microscope (JEOL) was used to analyse the morphological features of the sorbent materials before and after the adsorption studies. The crystalline nature of the Adsorbent was determined using X-ray diffraction (Bruker AXS -D8 Advance).

Adsorption Studies

The adsorption performance of pristine and modified carrot seeds was studied in batch experiments. Ni(II) ion concentration in water before and after adsorption was measured using atomic Absorption spectroscopy (4129DAAS) following acid digestion. The adsorption mechanism was interpreted using Langmuir/Freundlich/Temkin isotherms.

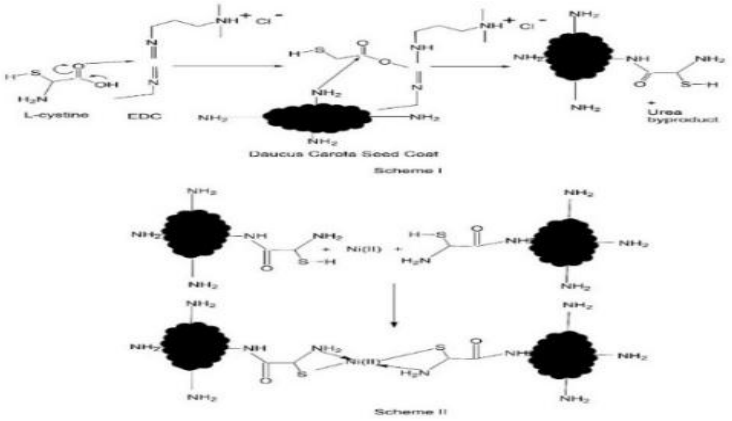


Fig.-1: Scheme for Modification of *Daucus Carota* Seed Powder and Mitigation of Nickel Ions from Water

RESULTS AND DISCUSSION

FTIR Studies

The spectra of the *Daucus Carota* seed show characteristic changes in the O–H/N–H (3600–3200 cm^{-1}) and main functional groups (1700–1500 cm^{-1}) after addition of L-cystine. Pure *Daucus Carota* seed powder showed characteristic bands at 3287, 2921, 1746, 1624, 1412, 1310, 1206, and 1033 cm^{-1}. Peak at 2921 cm^{-1} indicated aliphatic $–CH_2$ groups-asymmetric stretching (Fig.-2).

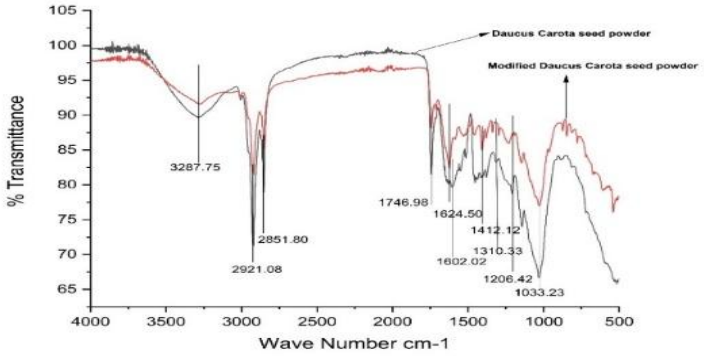


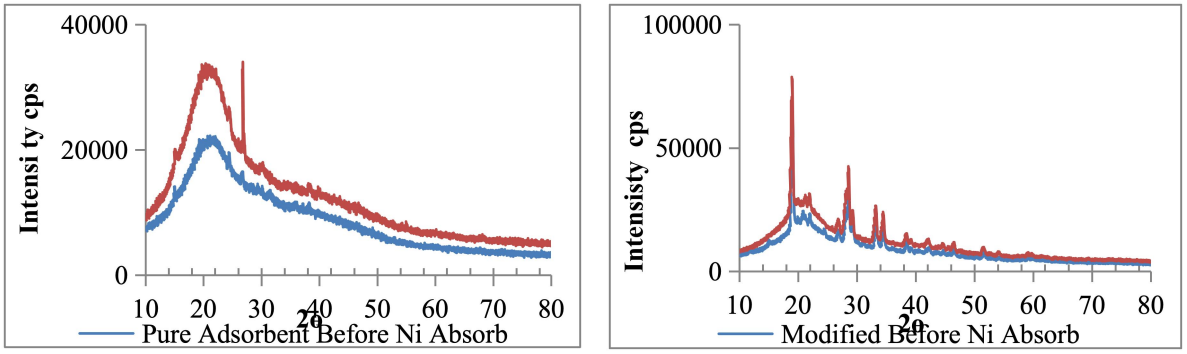
Fig.-2: FTIR Analysis of *Daucus Carota* Seed Powder and Modified *Daucus Carota* Seed Powder

Peak at 2851 cm^{-1} indicates symmetrical stretching of aliphatic $–CH_2$ groups. The carbonyl group in the adsorbent was confirmed by a peak at 1746 cm^{-1} and a peak at 1602 cm^{-1}, further indicating the presence of a conjugated system in the adsorbent. The peak at 1412 cm^{-1} is due to phenolic or carboxylate groups. The peak at 1310 cm^{-1} is due to amine groups. The peak at 1206 cm^{-1} is due to alcohol, ether, or ester groups. The peak at 1033 cm^{-1} is associated with polysaccharides or cellulose. After the addition of L-

cystine on the surface of *Daucus Carota* seed powder, slight shift-broadening and new peaks were observed in the IR spectrum. Pure *Daucus Carota* seed powder shows stronger and sharper peaks (especially at 2921, 1602, and 1033 cmv). However, these peaks have lower intensity in modified *Daucus Carota* seed powder due to changes in their environment and binding of the functional group with the L-cystine.

XRD Studies

The XRD of both the modified and pure *Daucus Carota* seed powder adsorbents is dominated by a broad reflection at 2θ ≈ 18–23°, attributed to the semi-crystalline lignocellulosic matrix and organic components (Fig.-3a). After adsorption of nickel ions from water, the *Daucus Carota* seed powder exhibits higher intensity and more defined features in this region, indicating interactions between nickel bivalent ions and surface functional groups, leading to partial structural ordering and a change in the phase composition. In both materials, a weak peak at 2θ ≈ 26–28° is assigned due to the disordered graphitic carbon, reflecting carbonaceous ordering within the adsorbent matrix (Fig.-3b). After modification of *Daucus Carota* seed powder, a few more peaks are observed due to changes in the crystallinity, particle size, structural ordering, and phase composition. The enhancement in the intensity is more pronounced in the modified *Daucus Carota* seed powder adsorbent after adsorption of nickel ions, suggesting more effective anchoring of nickel ions.



(a) (b)

Fig.-3: XRD Analysis of *Daucus Carota* Seed Powder and Modified *Daucus Carota* Seed Powder for The Removal of Nickel Ions from Water

Scanning Electron Microscope (SEM) Studies

SEM was used to investigate the structural changes of pure and functionalized *Daucus Carota* seed powder, providing insight into the physical effects of chemical modification on adsorption performance. The SEM image of DCS (Fig.-4a) shows irregular-layered/ plate like surface features with low porosity, and the SEM image of MDCS indicates significant variation in the surface features, as it shows a granular/highly fragmented porous structure (Fig.-4b). The increased porosity/surface area may help increase the adsorption capacity of the modified material.

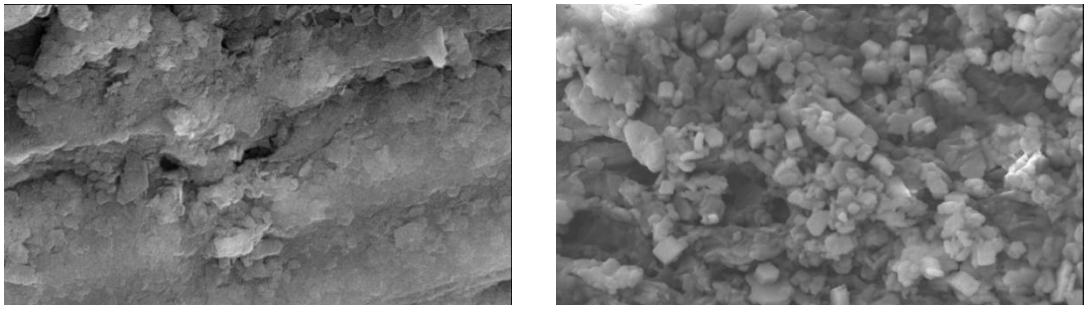


Fig.-4: SEM Analysis of a) *Daucus Carota* Seed Powder and b) Modified *Daucus Carota* Seed Powder

Adsorption Studies

Adsorption experiments were carried out at room temperature. In each experiment, a Ni(II) solution (specific concentration) was treated with a specified amount of adsorbent at a selected pH for a specified

contact time. The effect of pH in the range 1-8 has been studied at specific contact time/adsorbent dose/ initial concentration, and maximum removal was recorded at pH 8 for the pristine material and at pH 6 for the modified adsorbent, with a marginal increase observed for the modified material from pH 6 to pH 8. However, at higher pH values (more than 8), there was a significant decrease in adsorption. So, other experiments were conducted at pH 8 for the pristine material and at pH 6 for the modified adsorbent. Effect of adsorbent dose (0.25 to 2 g/L) was studied at a specific contact time, pH (6/8), and initial concentration (60 mg/L) and a significant increase in removal percentage was observed till 1g/L and no significant increase was observed after 1g/L. Effect of contact time (20 to 240 minutes) has also been studied at pH =6/8, dose = 1 g/L and initial concentration from 20 to 100 mg/L and adsorption equilibrium was achieved in 180 minutes. The removal of Ni(II) ions from water increased with increasing contact time for both adsorbents at all studied initial concentrations (20–100 mg/L). In all cases, a rapid uptake phase was observed during the first 180 min and equilibrium was achieved at approximately 180–240 min. The Effect of Ni ion concentration, ranging from 20 to 100 mg/L, was studied at a contact time of 180 min, pH of 6/8, an adsorbent dose 1g/L, and a temperature of 25–30 °C. The highest removal efficiency occurred at 60 mg/L, indicating an optimal balance between concentration driving force and adsorption site availability. Similar trends, where maximum percentage removal is achieved at intermediate concentrations, have been widely reported in Ni(II) adsorption studies using biomass-derived adsorbents, activated carbons, and clays.[28] The decline in percentage removal at higher concentrations is attributed to saturation of available adsorption sites and increased competition among Ni(II) ions. The equilibrium adsorption behaviour of Ni(II) onto the pristine and modified sorbents was further analysed using different isotherms.

Langmuir Adsorption Isotherm

The Langmuir isotherm is widely employed to describe adsorption on homogeneous surfaces (monolayer) and assumes no lateral interactions between adsorbed species, making it particularly suitable for systems in which adsorption continues until surface saturation is achieved. The Langmuir isotherm is the most extensively applied isotherm for evaluating adsorption capacity/adsorption performance in aqueous systems.[29] To quantitatively describe the equilibrium adsorption process, the Langmuir parameters were estimated as per the equation given below:

$$1/q_e=(1/q_{max}K_L)1/C_e+1/q_{max}$$

q_e (mg/g) refers to the amount of nickel ions adsorbed (per unit weight of sorbent material), and C_e (mg/L) represents the nickel ions equilibrium concentration. q_{max} (mg/g) expresses the maximum monolayer capacity of material for adsorption of Ni ions. Langmuir constant K_L (L/mg) indicates affinity. The further separation factor R_L can be calculated to indicate whether the isotherm is favourable.

$$R_L = 1 / (1 + K_L C_o)$$

C_o refers to the initial concentration of nickel ions. A value of the separation factor less than 1 indicates a favourable isotherm in the present case. The other parameters are listed in Table-2. For pristine material DCS, a plot of $1/C_e$ versus $1/q_e$ produced a slope of 0.2776 and an intercept of 0.0062 (Fig.-5a). From these values, the maximum monolayer adsorption capacity (q_{max}) was calculated as 161.29 mg/g and Langmuir affinity constant (K_L) was calculated as 0.022 L/mg. However, for MDCS, the plot of $1/C_e$ versus $1/q_e$ produced a slope of 0.1883 and an intercept of 0.0043 (Fig.-5b). From these values, the maximum monolayer adsorption capacity (q_{max}) was found to be 232.55 mg/g and Langmuir affinity constant (K_L) was calculated as 0.023 L/mg. The R^2 value confirms the validity of the Langmuir model for the present adsorption system. q_{max} values for DCS and MDCS indicate the adsorption performance of both materials for Ni(II) ions.

Langmuir constant (K_L) also confirms the favourable interactions between DCS/MDCS and Ni(II) ions. It has been reported that pristine materials have low adsorption capacity in comparison with their modified versions.[30-32] Adsorption capacity for MDCS is relatively higher than DCS in the present study, which confirms the increased interactions between the modified material and adsorbate. The obtained isotherm parameters also align with values reported in previous studies.[33-34]

Freundlich Adsorption Isotherm

Freundlich isotherms are commonly used to describe the adsorption of target species on a heterogeneous surface.[35]The standard-linear form of the isotherm can be expressed with the equation given below:

$$\ln q_e = \ln K_f + 1/n \ln C_e$$

where K_f is the Freundlich constant. n is related to adsorption intensity. The heterogeneity factor (n) value of 1–10 indicates favourable adsorption. The plots of $\ln q_e$ versus $\ln C_e$ (Fig.-5c & d) yielded Freundlich parameters K_f = 4.83 mg/g & 5.79 for pristine and modified material, respectively. The value of n more than 1 for both DCS and MDCS indicated favourable adsorption. Higher K_f of MDCS than DCS confirms the successful surface modification for Ni(II) ion uptake. Many reported studies have reported a similar change for modified materials.[30,32] Results indicate that both DCS and MDCS are suitable for Ni(II) uptake and can be selected based on the level of contamination.

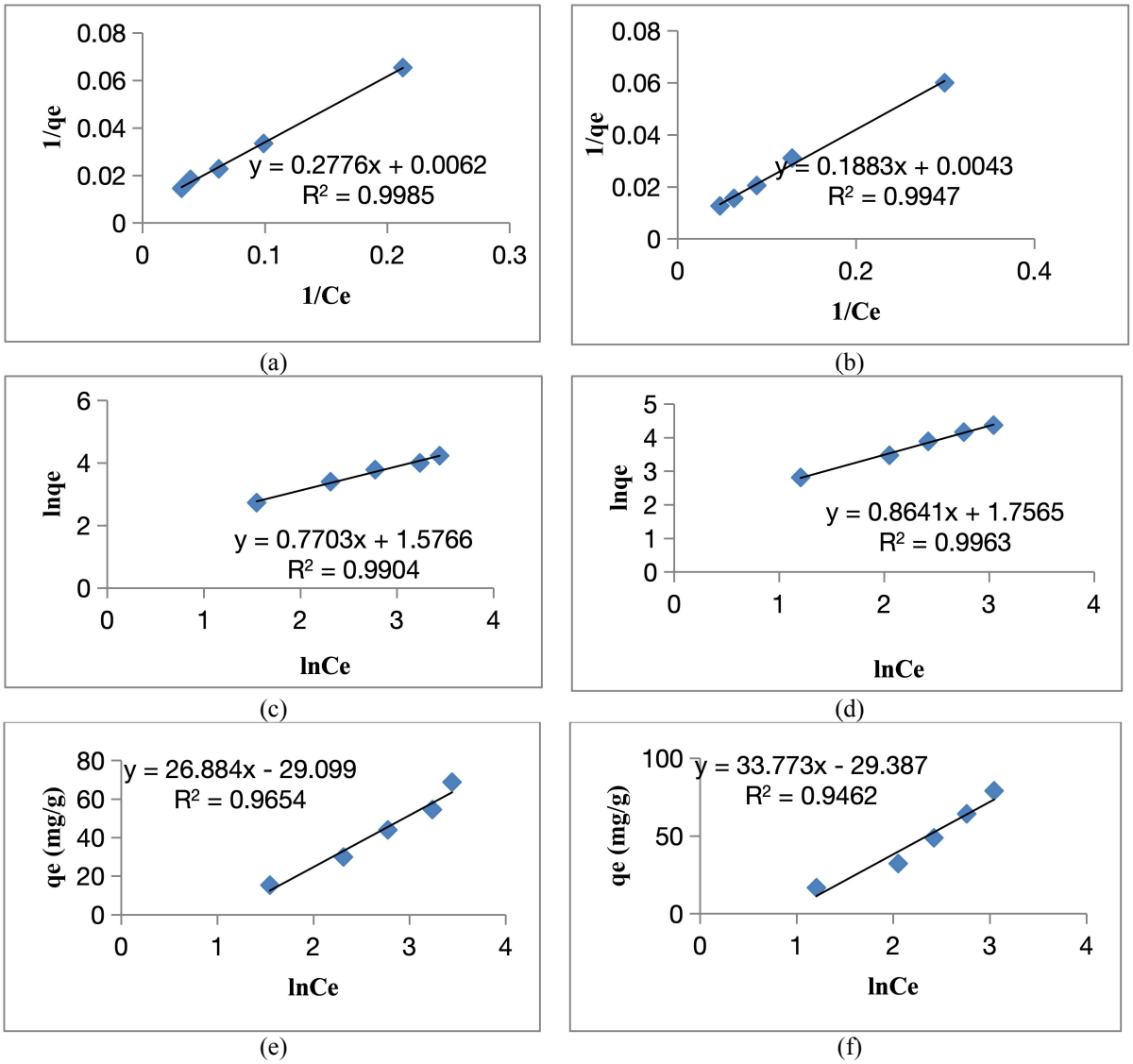


Fig.-5: (a) Langmuir Model for Pristine Adsorbent (b) Langmuir Model for Modified Adsorbent (c) Freundlich Model for Pristine Adsorbent (d) Freundlich Model for Modified Adsorbent (e) Temkin Model for Pristine Adsorbent (f) Temkin Model for Modified Adsorbent (pH = 8-Pristine 6-Modified; Adsorbent Dose = 1 g/L; Temperature = 25-30 ^{0}C; Contact Time = 180 Minutes).

Temkin Isotherm

The standard form of the Temkin model (linear) is:

$$q_e = B \ln A + B \ln C_e$$

Where B indicates the heat of adsorption (L/g) and A represents the Temkin constant. The value of the regression coefficient R^2, as shown in Fig.-5e and 5f, is lower than that of the Langmuir/Freundlich models, indicating that the model is poorly fitted to the present data.

Table-2: Isotherm Parameters

Adsorbent	Langmuir			Freundlich			Temkin	
	q_{max}	K_L	R^2	n	K_f	R^2	B	R^2
Pristine	161.29 mg/g	0.022	0.9985	1.298	4.83	0.990	92.157	0.9654
Modified	232.55 mg/g	0.023	0.9947	1.157	5.79	0.9963	73.35	0.9462

The sorbent capacity of various recently reported materials, for the removal of Ni ions from water, as given in Table-1, has been compared with proposed DCS and MDCS materials (Fig.-6). Eichhornia crassipes treated with water/NaOH showed the highest adsorption capacity at a contact time of 240 minutes and an initial concentration of 3500 mg/l, with an adsorbent dose of 3 g/L. The pristine *Dacus Carota* seed coat-based adsorbent was also found to be a promising, sustainable option for removing nickel ions from water, with a contact time of 180 minutes and an adsorbent dose of 1 g/L. Regeneration studies were also conducted for selected materials to assess the adsorbent's reusability. The DCS and MDCS were subjected to column regeneration using (30%) H_2O_2 prepared in (0.1 M) HNO_3 as the desorbing agent. After desorption, the material was further treated with 0.1 M NaOH, then washed with distilled water and dried. The dried adsorbent was collected and stored in airtight containers for subsequent use. The regeneration studies confirmed that both materials can be regenerated and reused for up to 3 cycles, with only a slight decrease in adsorption efficiency after each cycle (Fig.-7). Results confirm that these low-cost materials can be used as packing material in the column for real water samples and for wastewater treatment under specific conditions.

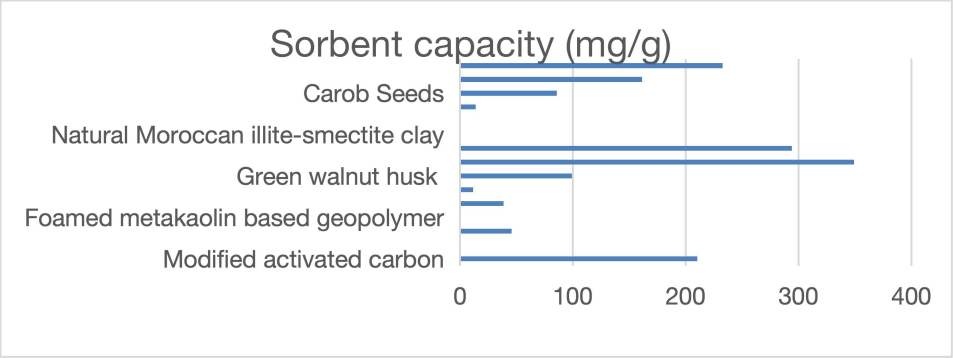
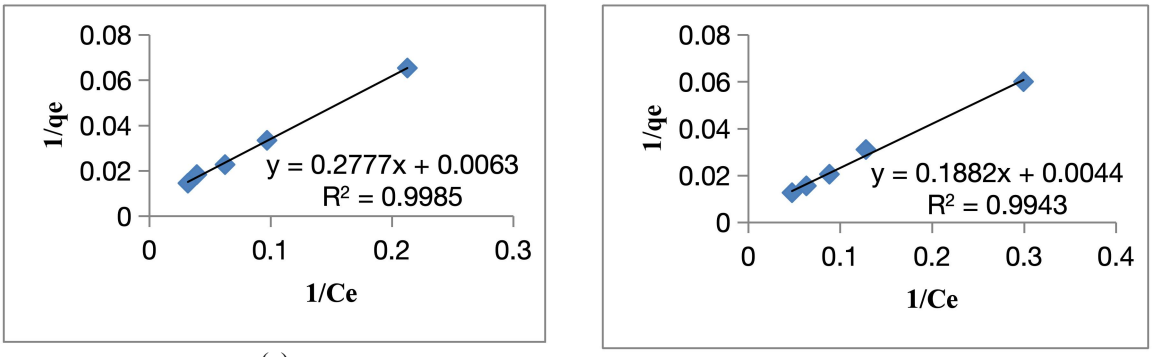


Fig.-6: Comparison of Sorbent Capacity of Reported Materials with DCS and MDCS



(a) (b)

Fig.-7: (a): Langmuir Model for Regenerated Pristine Adsorbent, (b): Langmuir Model for Regenerated Modified Adsorbent

CONCLUSION

Chemical modification of seed biomass significantly improves Ni(II) adsorption from aqueous solutions. The modified seed achieved higher removal efficiencies across initial concentrations of 20–100 mg/L, with faster initial uptake and higher equilibrium capacities than the unmodified seed. FTIR, SEM and XRD analysis confirms the modification of the seed biomass and is helpful to explain the adsorption performance of DCS and MDCS. The adsorption capacity of MDCS (232.55 mg/g) was found to be more than that of DCS (161.29 mg/g). Results confirmed that DCS and MDCS can be used to remove Ni(II) ions from water at pH 6-8 and room temperature. Further studies may explore the potential of these novel adsorbents for the simultaneous removal of other target contaminants from water.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean water and sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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