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## An Innovative Method for Removing Ni (II) from Contaminated Water that Uses Amino Acids as Facilitating Agents on certain adsorbents

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<b>KEYWORDS</b> Adsorption of Ni, Amino acid ligands, Montmorillonite, CNP, AC.	ABSTRACT: Nickel-amino Asparagine (A adsorption of 1 Montmorilloni formation of N AAs. The AAS by chelating li was 15.89% o Other measure procedure. Th demonstrated to desorption from	acid ligand complexes prepared us sp), Glycine (GLy), Lysine (Lys), Val Ligand-free Ni(II) and Ni-AAs comp te (MMT), Activated Charcoal (AC) Ni-AA complexes' was validated by S research endorsed that the adsorptio gands (2297.77 ppm from Ni-Val), at n MMT, 12.31% on AC, and 13.54% ements, such as XRD, SEM, and EDX ermogravimetric analysis (TGA) and that the stability of Ni-AA complexes m the surface of adsorbents is endothed	sing amino acids (AAs) Alanine (Ala), line (Val), and Hippuric acid (Hip) and the lexes investigated on selected adsorbents, , and Carbon Nano Particles (CNP). The comparing their FTIR spectra to those of n of Ni(II) from complexes' was increased nd by employing valine, the enhancement on CNP compared to Ligand-free Ni(II). X, were employed to assess the absorption I the differential thermal analysis (DTA) on adsorbents was stable up to 550oC, and ermic.

### **INTRODUCTION:**

The most vital element on earth is water<sup>1</sup> for a healthy ecosystem, agriculture, and for actions carried out by humans, and other living things. Water quality is declining because of population growth, civilization, residential and agricultural activity, and growing industrialization<sup>2</sup> and thus water pollution is a serious problem. All kinds of living things, homes, leisure activities, fishing, transportation, and other commercial pursuits are the source of water pollution. Heavy metals are elements with high atomic weights (63.5 to 200.6) that occurnaturally<sup>3</sup> and when present in water harm human health and aquatic ecosystems. The major heavy metals concerned are Hg, Pd, Cd, As, Cr, Ni, and Zn. Heavy metal pollution of water refers to hazardous heavy metals in water sources such as

rivers, lakes, and groundwater. They build up in sediments, plants, and aquatic creatures, causing bioaccumulation and biomagnification in the food chain and affecting life, which disrupts water ecosystems<sup>4</sup>.

Due to the characteristic qualities of nickel, it has a wide range of uses in industrial and commercial processes, including stainless steel<sup>5</sup> manufacturing, alloy formulations<sup>6</sup>, rechargeable batteries<sup>7</sup>, electroplating<sup>8</sup>, gas turbines<sup>9</sup>, surgical instruments<sup>10</sup>, and welding supplies<sup>11</sup>. Nickel is one of the crucial microelements for the human body's hormone balance and protein metabolism. However, exposure to nickel above the permissible concentration levels leads to various acute and chronic diseases<sup>12</sup>. The poisonous and non-biodegradable metal nickel<sup>13</sup>, enters water bodies when emitted by industrial 2914

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discharges, garbage disposal sites, and mine dumping, and results in water contamination. The contamination influences the development, procreation, and general health of the aquatic environment's organisms.

Nickel exposure at work can happen in the stainless-steel industry, in nickel mining, refining, and alloy manufacture, and it can cause kidney difficulties, bronchitis, lung fibrosis, asthma, lung cancer, and skin allergies<sup>14</sup>. WHO and the Bureau of Indian Standards (BIS) have recommended a maximum threshold of 0.02 mg/L for nickel in drinking water. WHO also established a lifetime provisional tolerated daily intake (PTDI) of 0.5 mg/Kg of body weight for everyone. PTDI levels above 0.5 mg/Kg over time may cause health problems associated with the respiratory, digestive, cardiovascular, and renal systems. The main reason for the presence of Ni<sup>2+</sup> ions in wastewater is their low affinity for adsorption and removal during wastewater treatment.

Generally heavy metals are removed from contaminated water using various techniques such as precipitation<sup>15</sup>, solvent extraction<sup>16</sup>, distillation<sup>17</sup>, adsorption<sup>18</sup>, ion membrane exchange<sup>19</sup>, water treatment<sup>20</sup>, phytoremediation<sup>21</sup>, and magnetic separation<sup>22</sup>. Among these, adsorption is the most practical and facile method for cheaply separating metals and metal ions from contaminated water.

According to literature, during the adsorption process, the chelating ligands such as triethylenetetramine (Trien)<sup>23, 24</sup>, EDTA<sup>25</sup>, and organic acids<sup>26</sup> are found to be considerably efficient in the removal/recovery of metal ions as corresponding metal complexes from bioleaching solutions than in the form of simple metal ions alone. Considering these findings, we are now intended to employ the easily and cheaply available amino acids (AAs) as effective complexing ligands for the improved removal/recovery of Ni(II) ions from contaminated water samples in the form of Ni-AA complexes adsorbed onto selected adsorbents. In general, AA ligands with Ni(II) are known to form stable complexes in aqueous and biological solutions for various applications.

This manuscript describes the efficacy of six amino acids and their derivatives (Alanine (Ala), Asparagine (Asp), Glycine (Gly), Lysine (Lys), Valine (Val), and Hippuric acid (Hip)) as complexing ligands for the efficient removal of Ni(II) ions from the contaminated water samples and subsequent adsorption on three selected adsorbents namely (i) montmorillonite (MMT), (ii) activated charcoal (AC), and (iii) carbon nano-particles (CNP). Ni-AA complexes are expected to display higher adsorption than the Ni(II) ions alone due to hydroxyl, carbonyl, and amino groups, which give various chemical and physical functionalities for adsorption onto selected adsorbents. Further, these complexes can exhibit diverse morphologies in terms of size, charge, and shape and makes them to interact with adsorbents in positive and negative ways. Consequently, different extents of adsorption may be achieved.

### **EXPERIMENTAL:**

Chemicals and materials were of analytical grade used in the research. 6.57 g of ultra-pure nickel sulphate [NiSO<sub>4</sub>.6H<sub>2</sub>O] was dissolved in 1000 mL of distilled water to create a 0.05N Ni(II) solution. It was then acidified with nitric acid to stop the hydrolysis process. Aqueous solutions of the six amino acid ligands (Ala, Asp, Gly, Lys, Val. and Hip) and nickel sulphate were combined in a 2:1 equivalent ratio to create the Ni-AA complexes, which were then left to set for five hours at room temperature. The FTIR spectra were recorded for Ni-AA complexes and correlated with the FTIR spectra of their amino acids.

To perform adsorption, 5.0 g each of MMT clay is combined with solutions of the six Ni-AA complexes and ligand-free Ni(II) and left for 48 hours for maximal adsorption. Also, for the adsorption of Ni-AA complexes and ligand-free Ni(II) on each of the AC and CNP adsorbents, the same procedure was followed. Using Whatman filter paper 1, precipitates containing Ni-AAs adsorbed on adsorbents, MMT, AC, and CNP were removed from the solutions, washed with 2% HNO3 and distilled water, and dried for ten hours in a hot oven at 60°C. All samples were analyzed using the AAS spectrometer (Thermos Scientific iCE 3300 AA01204006 V1.3O) by exposing them to the continuous signal of wavelength 248.3 nm for getting the quantity of Ni(II) adsorbed on the absorbents. Table 1 lists each of the calculated values in order.

Images of XRD (ASII-20 Intensity) measured at room temperature with Cu (as an anodic material wavelength of

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1.54056 A°) for Ni-Val complex and ligand-free Ni(II) on CNP taken to measure the extent of adsorption of Ni(II) and other elements of amino acids on adsorbents from Nival and ligand-free Ni(II). The SEM images, taken using 10 kV acceleration voltage at various magnifications, support the adsorption of Ni (II) complexes on adsorbents. DTA and TGA graphs were created using the DTG-60 Shimadzu (maximum temperature 1,100°C) to assess the stabilities and desorption temperatures of the Ni-AA complexes on adsorbents.

### **RESULT AND DISCUSSION:**

#### FTIR Spectra of Ni-AA complexes:

Examining and contrasting the FTIR spectra of free amino acid ligands, the formation of the Ni-AA complexes was reliable. The typical AA signals in the Ni-AAs' FTIR spectra are absent where they should be, while novel ones appear at 3200-3300 cm<sup>-1</sup>, 1632 cm<sup>-1</sup>, and 665 cm<sup>-1</sup>. There were no  $NH_{3^+}$  (asym str) signals at 3414 cm<sup>-1</sup> for glycine,

3099 cm<sup>-1</sup> for alanine, 3083 cm<sup>-1</sup> for valine, 3357 cm<sup>-1</sup> for asparagine, 3128 cm<sup>-1</sup> for lysine, or 3414 cm<sup>-1</sup> for hippuric acid in the FTIR spectra of Ni-AAs.The FTIR signals at 3084 cm<sup>-1</sup> for glycine, 2926 cm<sup>-1</sup> for alanine, 2988 cm<sup>-1</sup> for valine, 3000 cm<sup>-1</sup> for asparagine, 3047 cm<sup>-1</sup> for lysine, and 2938 cm<sup>-1</sup> for hippuric acid for asymmetric stretching of CH<sub>3</sub> are also absent. The FTIR spectra of Ni-AA complexes lack the CO stretching frequency signals for free amino acids between 1580 cm<sup>-1</sup> and 1750 cm<sup>-1</sup> of ligands. The signals at 3426 cm-1 of lysine are moved to a lower wavelength and altered as a band between 3200 cm<sup>-</sup> <sup>1</sup> and 3300 cm<sup>-1</sup> when coordinated with Ni(II). The OH stretching frequency signals related to valine, glycine, asparagine, and hippuric acid appeared between 2849 cm<sup>-1</sup> and 3169 cm<sup>-1</sup> in the FTIR spectra. It demonstrates how the nickel coordinated with carboxyl and amine donor sites of amino acids. These FTIR results show that the coordination of AA ligands to Ni(II) resulted in the production of Ni-AAs.



Fig. 1: FTIR spectra of Ni-AAs (Ni-Gly, Ni-Ala, Ni-Val, Ni-Asp, Ni-Lys, and Ni-Hip)

## Atomic Adsorption Spectroscopy (AAS)study of Ni-AA complexes:

Results of the adsorption of Ni(II) on three adsorbents viz., MMT, AC, and CNP in the presence and absence of AA ligands are communicated in Table 1.

Entries from Sl. No. 1-6 relates to the quantity of Ni(II) adsorbed onto the MMT when AAs are present and 7 AAs are absent. The findings showed that Ni(II) adsorbed onto the MMT from Ni-AAs was more than was observed in a solution containing pure Ni(II) ions. In the Ni-AA

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complexes, valine had the highest adsorption (2219.33 ppm) and is 15.89% more than Ni(II) without ligand.

Table 1. AAS data showing adsorption of Ni-AAs and ligand-free Ni(II) on MMT, AC, and CNP adsorbents.

SI. No.	Adsorbent	Ni-AA complex	Absorption of Ni in ppm.	Percentage of Ni-AA adsorbed on adsorbent compared to Ni(II)
1		Ni-Ala	1668.31	1.75
2		Ni-Asp	1623.28	0.39
3		Ni-Gly	1725.45	3.44
4	MMT	Ni-Lys	1723.99	3.40
5		Ni-Val	2219.33	15.89
6		Ni-Hip	1692.04	2.46
7		Ni(II)	1610.68	-
8		Ni-Ala	2109.42	10.99
9		Ni-Asp	2161.71	12.19
10		Ni-Gly	2108.45	10.96
11	AC	Ni-Lys	2119.1	11.21
12		Ni-Val	2167.04	12.31
13		Ni-Hip	1735.13	1.26
14		Ni(II)	1691.81	-
15		Ni-Ala	2010.16	6.91
16		Ni-Asp	2241.12	12.29
17		Ni-Gly	2176.72	10.85
18	CNP	Ni-Lys	2171.88	10.74
19		Ni-Val	2297.77	13.54
20		Ni-Hip	2110.87	9.34
21		Ni(II)	1750.28	-

The quantities of Ni(II) adsorbed on activated charcoal (AC) shown are in Table 1, entries from Sl. No. 8–14. The ligand-free Ni(II) solution had the lowest Ni(II) adsorption (1691.81 ppm). Hippuric acid ligand did not alter the adsorption significantly (1735.13 ppm). But when AA

ligands were present, significant Ni(II) adsorption was seen (Table 1, entries 8–12). In particular, the adsorption of the Ni(II)-val (2167.4 ppm) and Ni(II)-Asp (2161.71 ppm) systems was higher, or about 12.31% more than that of the ligand-free Ni(II) system.

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The amount of Ni(II) adsorbed onto CNP, as seen in entries 15-20 of Table 1, the usage of AAs and hippuric acid has demonstrated a positive effect on increasing the adsorption of Ni(II). Ni-valine had the highest adsorption of these (2297.77 ppm). As anticipated, the ligand-free system showed the lowest Ni(II) adsorption (1750.28 ppm), meaning that without a ligand, Ni(II) adsorbed was at a rate that was approximately 13.54% lower than that of the Ni-valine complex.

Figure 2 depicts the broad pattern of the three adsorbents' elimination of Ni(II) ions when AAs were present. Quantity-wise, the MMT, a non-carbon clay material, has a low adsorption capacity except for the Ni-valine complex. The observation was that AC has an adsorption capability between CNP and MMT. In general, CNP has shown a greater capacity for the adsorption of NI-AA compounds. The CNP adsorbent's nanoscale carbon particles have a high surface area and porosity, contributing to their higher adsorption capacity.



Fig. 2: Comparison of adsorption of Ni from Ni-AAs of amino acids.

Figure 3 shows the effectiveness of AAs for the simple adsorption of Ni(II) onto three adsorbents compared to a ligand-free sample setup. All AAs, except alanine, improved the adsorption of Ni(II) onto CNP. Among all the AAs used in the studies, valine showed the highest adsorption on the MMT, AC, and CNP adsorbents. Valine and asparagine demonstrated higher adsorption, followed

by glycine, lysine, alanine, and hippuric acid displayed the lowest adsorption. The big non-polar benzene ring of hippuric acid may be less alluring to adsorbents, resulting in lower adsorption. Comprehensively, all three of the chosen adsorbents showed the lowest adsorption on the ligand-free Ni(II) containing water sample.

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Fig. 3: Adsorption of Ni and Ni-AAs on adsorbents Mont, AC, and CNP.

### XRD and SEM studies:

The type complexes generated between CNP and Ni(II) and the complex between CNP and Ni-Val were studied using X-ray diffractograms, and the peaks in these data indicated the development of crystals. Displaying the

pattern of the height (at  $2\Theta = 26.4861$ ), which is 592.28 counts, Figure 4 demonstrates the Ni-CNP crystal's existence. As a result, 431.13 counts were determined for Ni-Val-CNP (at  $2\Theta = 26.4892$ ), indicating crystal formation.



Fig. 4: XRD pattern of Ni(II) adsorbed on CNP and Ni(II)-Val complex on CNP.

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The observed white patches in the SEM images correlate to adsorbed Ni (Figure 5 a) and Ni-AA complexes (Figure 5 b) on CNP. Furthermore, the Ni-AA complex on CNP has occupied a larger surface area than just basic Ni adsorbed on CNP. This finding shows that metal-ligand complexes have more adsorption capacity than metal ions alone.



Fig. 5: a) SEM of Ni on CNP; b) Ni-AA complex on CNP

Besides, the EDX spectrum of Ni(II) and Ni-Val complex deposition on CNP (Figure 6 a and b) provides a piece of evidence for the adsorption of Ni on CNP. The EDX spectra revealed 2.79% ligand-free Ni(II) and 32.54% of the Ni-Val complex adsorption on CNP. There is an enormous increase in adsorption from metal-ligand complex to metal ion.



Fig. 6: EDX spectrum of a) Ni(II) on CNP; b) Ni-Val deposition on CNP adsorbent.

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### Thermo gravimetric analysis of Ni-AA complex:

Thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) has proven to be powerful technique to measure the physical and chemical changes that occur when ligands bind to metal ions and adsorb the complexes to adsorbents. Figure 7 illustrates the two steps of the decomposition/desorption of Ni-AA from the CNP adsorbent. The TGA profile has two curves related to decomposition/desorption. The first curve is observed at 345°C for a loss of 32.204% weight, while the second is at 559°C for 41.526% weight of the Ni-Val complex adsorbed on CNP. The outcomes showed that adsorbed Ni-AA complexes are thermally stable up to 550°C. This kind of adsorption, therefore, is used in pyrometallurgical processes to remove metal/metal ions from contaminated water and soil by employing ligands as chelating agents.

In differential thermal analysis (DTA), curves with peaks at the locations of corresponding mass losses suggest that desorption of Ni-AA complex from the surface of CNP is exothermic, i.e., accompanied by energy losses.



Fig. 7: TGA and DTA coupled analysis showing decomposition of Ni-AA from CNP.

#### **CONCLUSIONS:**

Using CNP, AC, and MMT adsorbents in the presence of specific amino acids as complexing ligands/agents is proved to be an efficient approach for the removal and recovery of Ni(II) from polluted water samples was developed. Compared to ligand-free Ni(II) samples, chelating AAs were better able to create a stable Ni-AA complex in water samples, which increased the concentration of Ni(II) on adsorbents. The FTIR spectrum data confirms the coordination of Ni(II) and amino acids to produce Ni-AA complexes. Compared to AC and MMT adsorbents, the nano-sized carbon particles in CNP have a

higher surface area, are more porous, and exhibit good adsorptive ability.

Especially in the case of the Ni-valine complex, a branching valine structure may be of a size that is adequate for the active sites of the adsorbents, leading to higher adsorption, as demonstrated by the higher rates of 15.88% on MMT, 13.54% on CNP, and 12.40% on AC compared to ligand-free Ni(II). SEM pictures made it abundantly evident that the Ni-Val complex covers a greater region on the surface of CNP than Ni(II). EDX profile also verified that the Ni-Val complex occupied 32.54% of the surface area on CNP, and it is only 2.79% by Ni(II). The TGA and DTA experiments showed that adsorbed Ni-AA complexes

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are stable up to 550C. The results of this study will surely pave the way for the strategic synthesis of metal-ligand complexes that promote heavy metal adsorption from contaminated water in the design of new remediation techniques.

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