A Brief Study of Various Adsorbents for Removal of Ni Metal Ions from Waste Water

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Abstract **- Discharge of waste water from industries is a major cause of heavy metal pollution of water. A large number of industries like electroplating, mining, paints and pigments, fertilizers, textiles, refining, smelting etc. discharge waste water containing heavy metals like Ni, Cd, Co, Zn, Hg, As, Cu, Cr, Pb etc. into water bodies. These metals have adverse effects on the health of humans, animals, plants and other organisms. Heavy metal pollution of water is becoming a major issue of public health and environment as these are non-biodegradable, toxic and carcinogenic. In the past few decades various researchers have employed a number of methods like membrane separation, ion-exchange, solvent extraction, precipitation, electrochemical process and adsorption for the removal of various heavy metal ions from waste water. Among these methods adsorption is found to be highly effective as it is cost–effective, easy to apply. Various adsorbents of different origin are used by different researchers like biomaterials, clays, minerals, resins, synthetic adsorbents, industrial by-products and many more for the efficient and effective removal of heavy metal ions from waste water. The present paper gives a study of the various adsorbents used for removal of Ni metal ions from waste water.**

***Keywords*-** **Adsorption, Adsorbents, Waste Water, Heavy metals, Ni.**

I. INTRODUCTION

Growth of industries has lead to a number of problems in environment. Out of them water pollution is a major concern all over the world. Industries discharge waste water directly into aquatic stream which contains lot of chemicals, both organic and inorganic, colored substances, heavy metals and many more. Heavy metals are the main cause of water pollution as these are not biodegradable and accumulate in food chain. These are highly toxic and many are carcinogenic [1]. Humans are at the top of food chain. So, they are the most adversely affected by heavy metal pollution. Direct discharge of these metals into aquatic ecosystem poses a great risk to aquatic organisms even at very low concentration.

Most common and harmful heavy metals like Al, Pb, Ni, Co, Cr, Cd etc. are discharged by various industrial activities like mining, metal plating, welding, battery manufacturing [2].

Ni is a heavy metal used in a large no. of industries like electroplating, Ni-Cd batteries manufacturing, forging, mining[3] ,coal and oil-burning power plants, etc.[4].Ni is 22nd most abundant element by weight in earth's crust [5]. In drinking water, maximum contamination limit set up by United States Environmental Protection Agency is 0.1 mg/L. As per the recommendations of WHO maximum admissible concentration of Ni (ii) ions in aqueous solution is 0.02 mg/L[6] .But metal ions are discharged into water bodies at much high concentration than the permissible limit by industrial activities[7] . High concentration of Ni causes lungs and bone cancer . Exposure to Ni results in skin irritation, damage to nervous system and mucous membrane [9], weakness, dizziness [10]. So, there is need to remove Ni metal ions from effluent waste water by employing treatment methods.

II. Different Methods for Ni Removal

The most commonly used physico-chemical methods are chemical precipitation, ion-exchange [11], solvent extraction, adsorption, membrane separation [12], electrolytic process and reverse osmosis etc. Most of these methods suffer from various disadvantages like high cost, generation of toxic sludge which requires disposal, incomplete removal of metal ions, high energy requirement [13]. Although ion-exchange resins have high adsorption capacity but these are pricey and sophisticate [14]. Similarly electrolytic process requires high energy and is not cost effective. Chemical precipitation of metals as hydroxides, carbonates, sulfides is usually used [7] but it requires large tanks for settling of sludge generated [14]. Hence there is urgent need for the development of a method which is highly cost effective, easy to implement, requires less processing, eco-friendly [15]. Researchers have developed adsorption as highly cost-effective and efficient method for uptake of heavy metal ions from effluent water. Process of adsorption employs a number of different adsorbent from various sources to achieve the goal in the field of heavy metal removal. Various adsorbents employed are biopolymers, synthetic adsorbents, plants and animals waste micro-organisms (algae, bacteria, macrophytes, and fungi), resins, industrial by-products, clays, minerals, activated carbons.

Process of adsorption has many merits like low-cost of adsorbents, ease of operation, lesser operational cost, abundant availability of adsorbents, reuse of adsorbent after regeneration in many cases, environment friendly, utilization of waste from industries, domestic and biological activities[7]. It is considered as efficient method as it involves removal of metal ions present even in low concentration and recovery of metal ions from loaded adsorbent [16].

III. Adsorbents Used for Ni metal Ions Removal

Commonly employed adsorbents are activated carbon, agricultural sorbents, industrial by-products, Zeolites, resins, micro-organisms, Nanomaterials and synthetic adsorbents.

A. *Activated Carbon*

Physical and chemical properties of AC depend on its pore size, no. of surface active functional groups and distribution of pores. These properties can be improved by various modifications of AC by use of activating agents which helps to improve adsorption capacity [7]. AC from almond husk has been used without and modification with H_2SO_4. It shows 97.8% Ni (ii) removal at 700^0C carbonization temperature, pH 5, adsorbent concentration of 5g/L and with addition of H_2SO_4 [17]. At this temperature, adsorbent has large internal surface area and maximum porous structure. Ni has also been removed by using different grades of both raw granular AC and chemically oxidized AC [18]. Different types of activated carbons have different surface characterization. Raw F-200 AC has slightly large surface area than AC oxidized F-200. GAC has more adsorption capacity for Ni^{2+} ions. This has been explained by increase in number of surface functional groups [18]. Similarly phosphoric acid activated carbon has been prepared from oil palm and coconut shell. Adsorption capacity of acid AC has been compared with commercial AC. Studies reveal that PSW-P-ad-500 (derived from palm shells semi dried impregnated feedstock) shows high adsorption capacity for Ni^{2+} ions as compared to other activated carbons [19]. The order of maximum monolayer adsorption capacity q_{max} is given as CAC << PSW-P-500 < CPW-P-500 << PSW-P-ad-500 with q_{max} values as 3.18 << 10.83 <12.18 <<19.61 mg/g. Moreover it has been shown that prepared activated carbon performs significantly in low conc. range while commercial activated carbon (CAC) performs in high conc. range [19]. Further studies involve use of AC cloth (ACC) for uptake of Ni metal ions from effluent water [20]. ACC are commercially available and desorption studies are possible with HCl solution. Two types of ACC- CS1501 and RS 1301 have been used in studies by the researchers [20]. Adsorption capacity results show that CS1501 is better adsorbent than RS1301. Adsorption on ACC occurs by ion-exchange mechanism and carboxylic groups on the surface are involved in adsorption process. AC from Thespesia populnea bark prepared by treatment with conc. H_2SO_4 in weight ratio 1:1 has been used for efficient removal of Ni metal ions [21]. Batch adsorption studies have been carried out to study the effect of adsorbent dose, initial metal ion conc., change in pH, presence of other ions. Thermodynamic parameters indicate the process to be endothermic and spontaneous. Desorption of adsorbent has been possible with HCl with more than 90% removal of adsorbed metal ions.

B. Agricultural wastes as adsorbents

Various agricultural based materials like stem, root, leaves, bark, peels, husks, beans, straw and fibres have been employed in the past decades by researchers as low cost adsorbents for effective uptake of heavy metal ions from waste water. Numerous studies on Ni metal ions removal are available in literature based on plant materials as adsorbents. These materials contain cellulose, lignin, hemicelluloses, sugars, proteins, lipids, starches and other compounds. In these compounds many functional groups like hydroxyl, carboxyl, amino, amido, phenolic, sulphdryl, acetamido, esters are present [22, 23]. These functional groups remove Ni metal ions either by ion-exchange or metal complexation.

1) Peels as adsorbent:

Orange peel contains high content of cellulose, pectin, hemicelluloses and lignin. It has been used as low cost adsorbent for Ni metal ion uptake [8]. Batch adsorption studies of orange peel were carried out to study the effect of change in pH, temperature, initial conc. of metal ions and dosage of adsorbent on removal of Ni (II). Experiments were performed in pH range 3-5. Adsorption increase with pH from 3 to 5 and maximum adsorption occurs at pH 5. At 25^0C maximum adsorption capacity of Ni (II) was 62.3 mg/g [8]. In comparison to this orange peel modified by graft copolymerization, shows increased adsorption with increase in pH from 2 to 5.5 [24]. Acid and alkali treated banana peel has been employed for studies on Ni (II) adsorption. Maximum adsorption capacity reported by studies is 6.88mg/g. Nitric acid and NaOH were used for modification of peel [25].

2) Husks, straws and bran as adsorbents:

Studies on unmodified and modified coconut husk have been carried out. A comparison of adsorption studies on NaOH and HCl modified coconut husk with unmodified coconut husk reveal that NaOH modified adsorbent has minimum adsorption for Ni(II) followed by unmodified adsorbent and maximum adsorption by HCl treated adsorbent. All the adsorbents have high adsorption of 90-99% at pH 8-9[26]. Straw from wheat has been used for removal of Zn (II) and Ni (II) ions in single and binary metal solutions. Reports show that adsorption of Ni (II) is decreased in presence of Zn (II) in binary systems [27]. Adsorption studies on bran, a by-product of wheat and rice crops for uptake of Ni (II) have reported that amount of Ni (II) adsorbed by wheat bran is 12.0 mg/g at pH 5 [28]. As compared to wheat bran, phosphoric acid modified rice bran shows maximum adsorption capacity of 46.51 mg/g at pH 6 [29].

3) Saw dust as adsorbent:

Saw dust is produced in large quantities as solid waste at saw mills. It is a cost effective, highly available and renewable source. Saw dust of oak (Quercus coccifera) tree treated with HCl has been used for effective removal of Ni metal ions [9]. Modification with HCl results in increase in lignin content of material which improves the adsorption capacity of adsorbent for heavy metals. Studies on effect of pH change on adsorption reveal that removal percentage of Ni (II) increase with increase in pH with maximum removal efficiency of 82% at pH 8. Studies on adsorption isotherm show that data fit well with Langmuir and Dubinin- Radushkevich adsorption isotherms. Researchers have also used sawdust of walnut for removal of Ni (II) from effluent water [30]. Maximum adsorption capacity reported in studies for Ni (II) by sawdust of walnut has been 6.43 mg/g. Walnut sawdust has been used after modification with HCHO in sulphuric acid.

4) Leaves as adsorbent:

Leaves of neem have been employed as a novel adsorbent for Ni (II), Cu (II) and Zn (II) ions uptake in batch experiments [31]. The percentage of Ni (II) ions removed is 68.75 with adsorbent dosage of 1.0 g at pH 7. As compared to this, studies on use of neem leaf powder (Azadirachta indica) as biosorbent for Ni (II) ions have shown 92.6% adsorption at pH 5 for adsorbent dosage of 4g/L [32]. Large adsorption capacity of Azadirachta indica has been due to presence of functional groups like carboxyl, hydroxyl, carbonyl etc. on its surface as reported by FTIR measurements. Batch adsorption studies have also been carried out for potential removal of Ni (II) ions using leaves of Solanum elaeagnifolium (Silver leaf nightshade) desert plant [33]. Results reveal that maximum binding of metal with native Solanum elaeagnifolium take place at pH 5 within 10-15 minutes of contact time and adsorption capacity is 6.5mg/g. Chemical modification of Solanum elaeagnifolium with NaOH enhances adsorption capacity as NaOH converts methyl esters present in plant tissues to carboxylate ligands by hydrolysis reaction. Recovery of metal ions is also possible by treatment with dilute acid.

5) Stem/bark as adsorbents:

Shoot biomass of Medicago sativa (alfalfa) has been employed in batch experiments for adsorption studies of Ni (II) in multi- metal solutions [34]. Binding capacity has been reported as 49.2µmol/g. Desorption studies were carried out with HCl solution and involve 90% or more recovery of bound metal ions. Column experiments have also been carried out with silica immobilized shoot biomass. Research results show that binding occurs by electrostatic interaction of metal ions with carboxyl groups present on adsorbent. Bark of Moringa oleifera was used for adsorption studies and it showed maximum adsorption capacity of 30.38 mg/g at optimum pH 6 for Ni metal ions [35]. Removal percentage was very low under highly acidic conditions as at low pH; H^+ ions compete with metal ions for adsorption sites. Adsorption increased with increase in pH from 3 to 6 and then decreased in pH range 7 and 8. Stem, leaves and roots of Larrea tridentata (Creosote Bush), a desert plant has been employed in adsorption studies of Ni metal ions from aqueous solutions [36]. Binding capacity experiments have shown that leaves have more adsorption capacity than stems and roots. Upto 99% desorption have been reported with HCl. Maximum adsorption capacity reported in studies on stems, leaves and roots has been 4.60, 5.90 and 5.60 mg/g respectively.

Many other agriculture based materials have been reported in adsorption studies like palm fruit fibre for Ni metal removal. It showed about 87% removal of Ni (II) ions [37]. Another biosorbent used is Cassia fistula (Golden Shower), an ornamental tree [38]. Various parts of plants have been selected for studies and maximum adsorption capacity reported in studies is 145.29-188.40mg/g. Saccharum bengalense plant, a novel green adsorbent contains cellulose and it removes about 87%

Ni metal ions [4]. Research has been carried out on dead and living aquatic fern Azolla filiculoides for adsorptive removal of Ni (II) ions. Maximum adsorption capacity by alkali and $CaCl_2/MgCl_2/NaCl$ (2:1:1 molar ratio) activated fern for Ni^{2+} ions was 1.365-1.198 mmol/g while non-activated fern showed adsorption capacity of 1.212-0.931 mmol/g in temperature range 283-313K [39]. Kinetic and thermodynamic studies on modified black carrot (Daucus carota) residues reveal that process of adsorption follows Lagergren first order rate equation and is endothermic in nature. Maximum adsorption capacity reported is 5.745 mg/g [14]. Freshwater macrophytes Alternanthera philoxeroides (alligator weed) was used for sorption of Ni (II) ions with maximum adsorption capacity of 9.73mg/g [15]. Environmental stability and adsorption efficiency of Zea mays cob powder has been increased by acetylation, succination and graft co-polymerization [40]. The material showed regeneration cycles from 3-5. Maximum sorption of Ni^{2+} has been reported as 71.98% in single metal solution and 64.03% in multi-metal solution. Extensive studies have been available on other plant materials as adsorbents in literature.

C. Industrial waste of agricultural origin as adsorbent

A number of adsorbents has been obtained from agro-industrial waste for Ni metal ions removal from waste water. Grape stalk from wine producing and exhausted coffee from coffee manufacturing industry was used for effective removal of Cu and Ni metal ions [41]. Research was carried out in presence and absence of strongly complexing agent EDTA. Metal uptake was pH dependent. It has been investigated that grape stalk was more efficient for uptake of metal ions in presence and absence of EDTA while exhausted coffee was less effective in presence of EDTA. Heavy metal recovery over 97% was achieved with HCl. Tea industry waste was used in fixed bed studies of Ni uptake [42]. Research was carried out as a function of liquid flow rate, bed height, pH, particle size of adsorbent, initial metal ion concentration. Waste arca shell marine biomass from fishing industry was used as adsorbent for Pb, Ni, Cu and Co metals uptake from aqueous solutions [10]. Maximum uptake of Ni has been reported as 9.86±0.17mg/g under optimum conditions of pH, adsorbent dosage, contact time, initial concentration of metal ions.

D. Synthetic materials as adsorbents

Researchers have synthesized a no. of synthetic materials for adsorption purpose. Poly [N-(4-[4-(aminophenyl)methylphenylmethacrylamide])] was synthesized from Poly-methyl methacrylate and 4,4'-diaminodiphenylmethane. It was characterized by FTIR, SEM and EDX. Regeneration of material was carried out with HNO_3 [2]. Non-ionic hydrogels were synthesized by radical homopolymerization of N-vinyl-2-pyrrolidone (VP) or by radical copolymerization of N-vinyl-2- pyrrolidone(VP) with methylacrylate. In the synthesis a microinimer was used as cross-linker and initiator [43]. Swelling properties, average molecular weight between two consecutive crosslinks and metal binding properties have been investigated. Regeneration studies by acid hydrolysis were also carried out. Another synthetic material used as adsorbent is thermosensitive copolymer prepared by free radical copolymerization of N-n-propylacrylamide and (dimethoxyphosphoryl) methyl2-methylacrylate followed by hydrolysis. Maximum adsorption capacity obtained at pH 7 and 20^0C was 0.6mmol/g [44].

E. Micro-organism based adsorbents

Adsorption studies using algae, fungi, bacteria and yeast micro-organisms as biosorbent have gained importance due to their cost effectiveness and ability to accumulate heavy metals. Chemically modified brown algae Laminaria japonica has been reported for Ni (II) uptake from waste water [45]. Modifications were carried out by cross linking with epichlorohydrin, by oxidation with $KMnO_4$ or glutaraldehyde. Epichlorohydrin modified adsorbent showed higher adsorption capacity as compared to raw biomass, $KMnO_4$ and glutaraldehyde treated adsorbent. Rhizopus

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delemar, a fungal biomass has been reported for bioaccumulation of Ni (II) and Cu (II) ions during the growth phase of fungus [46]. Biosorption of Ni (II) ions onto inactive Saccharomyces cerevisiae has been investigated as a function of various parameters in batch system [47]. Maximum metal uptake has been 46.3mg/g for Ni (II) ions. Adsorption is physical, spontaneous and exothermic in nature.

F. Nanomaterials as adsorbents

Nano crystalline hydroxyapatite has been investigated for adsorption of NI (II) ions from waste water [48]. Adsorption depends on charge density and hydrated ion diameter, Hydroxyapatite has low water solubility, high stability and low cost. Maximum adsorption capacity for Ni (II) ions has been reported as 40.00mg/g. In another studies sodium dodecyl sulphate coated magnetic nanoparticles were used for removal of Cu, Ni, Zn metal ions from water samples [49]. Adsorption is very fast under optimum conditions and maximum adsorption capacity for Ni(II) ions was reported as 41.2mg/g. Desorption studies have been carried out with methanol and material can be reused even after three adsorption- desorption cycles. Adsorption studies on conc. HNO_3 oxidized carbon nanotubes were carried out in single, binary, ternary and quaternary systems for Ni, Cu, Zn and Cd from aqueous solutions [50]. In single and binary systems, metal ions were adsorbed in order of $Cu^{2+} > Ni^{2+} > Cd^{2+} > Zn^{2+}$. Amino functionalized magnetic grapheme composite was prepared by composting grapheme sheet with ferroferric oxide [51]. It showed sorption capacity of 22.07mg/g for Ni (II). Material can be recovered from water with magnetic separation at low magnetic field within one minute.

G. Ion Exchange resins as adsorbents

Ni metal ions from waste water have been removed by use of ion exchange resins like Dowex HCR S/S [52] and D301R [53]. Batch adsorption studies were performed to examine the effect of dosage of resin, pH change and contact time. Results of study were tested for Langmuir, Freundlich and Tempkin adsorption isotherms. About 98% adsorption efficiency was observed for Dowex HCR S/S resin. Adsorption capacity of D301R has been reported as 84.3mg/g. Regeneration ability of resins further adds economic benefits to adsorption studies.

IV. Conclusion

A review of literature studies on adsorption of Ni metal ions from aqueous solutions has shown that various adsorption mechanisms are involved in adsorption. Various adsorption parameters have been taken into consideration while studying metal uptake like contact time, pH change, temperature, particle size, Initial conc. of metal ions etc. Adsorption kinetics follows either pseudo-first order or second order in most of studies. Obtained data was subjected to equilibrium isotherms like Langmuir, Freundlich , D-R isotherm and Tempkin. Thermodynamic parameters like enthalpy change, gibb's free energy change and entropy change were investigated to predict spontaneity, endothermic or exothermic nature of process. There is lack of data on regeneration and reusability of adsorbents in many cases. Research studies should be extended to real industrial effluents rather than stock solutions in laboratory.

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