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Functionalized Novel Nano Composites in Ni(II) Removal from Aqueous / Effluent Media

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Abstract: ***Nanohydroxy apatite – Azaridachtaindica nutshell composite material was prepared via co-precipitation method. The product was characterized by FT-IR and Atomic force microscopy. The Ni(II) removal capacity of the synthesized novel material is investigated by employing batch mode to verify the sorption efficiency under optimized conditions viz.,11 mg/L initial concentration of Ni(II), 21 minutes agitation time between sorbent/sorbate molecules and 50mg dosage of the nanocomposite. The results of the experiment registered a significant increase in the sorption nature of nanohydroxy apatite composite of the chosen nutshell against the chemically modified Azaridachtaindica nutshell where 450 mg dosage is employed under similar operating conditions. The successfullness of the nanomaterial in trapping Ni(II) ions is extended to field levels through the assessment of effluents collected from electroplating industry suggesting that nano composited biomass of Azaridachtaindica nutshell is an efficient bioaccumulant.***

Keywords: adsorption, nanocomposite, characterization, *Azaridachtaindica* nutshell, effluent

1. Introduction

Water is one of the most indispensable and unique substance, because it can naturally renew and clean itself, by allowing pollutants to settle out through the process of sedimentation. Ground water is highly valued because of certain properties not possessed by surface water[1]. It is required by industries for various reasons such as cooling, steam generation, heat exchange, gas scrubbing, washing of solids, surface rinsing etc. Today, the water is polluted with hundreds of toxins, heavy metals and impurities. This is a major problem in the global context. It has been suggested that it is the leading worldwide cause of deaths and diseases which accounts for the death of more than 14000 people daily.[2]Such toxic metals are removed from the aqueous and industrial streams using various sequestration techniques. The cost-effective, being the adsorption method which employs chemically modified agriculture materials as biosorbents for the removal. Such modification of agricultural waste materials can extract soluble organic compounds and enhance their chelating efficiency.

Due to dearth of information on the adsorption of heavy metals on Nano-modified sorbents, this modification procedure was adopted and applied to the selected agricultural sorbent.

2. Materials and Methods

Neem tree, botanically known as *Azadirachta indica*, belongs to the family Meliaceae. The tree is tall, fast growing and is known for its drought resistance [3].Neem seed nut shell husk were thoroughly washed well with double distilled water and dried in the air. The dried shells were powdered, sieved and the fractions measuring from 0.18 to 0.71 mm were prepared and designated as AINS.

A one-step co-precipitation method[4] was adopted to prepare theNano- composite of 0.18mm AINS-Nanohydroxyapatite. The white nano hydroxyapatite powder obtained through the method was washed well with double distilled water several times to bring the pH to 7 and later dried under normal conditions. The dried nano powder was then mixed with 0.18 mm of AINS in the ratio of 1:1 by adopting the one step co-precipitation method. pH adjustments were done using acetic acid and NaOH solutions. The obtained nano hydroxyapatite-adsorbent composite was then dried well and utilized for the specific studies. The Nanohydroxyapatite – *Azaridachta indica nut* shell composite is mentioned as NAINSC.

Known weights (50mg-200mg) of the adsorbent were taken in 250 ml Erlenmeyer flasks at room temperature (250C) and equilibrated with 50 ml of Ni(II) solution of known concentrations of 11 ppm was prepared from its sulphate salt [$Ni(NH_4)_2SO_4.9H_20$]. Fresh dilutions were used for each study. The pH values (3, 5, 7, 9) of the solutions were adjusted with 0.1M HCl /0.1M NaOH solutions respectively. The flasks were then agitated in an orbital mechanical shaker at 120 rpm at predetermined time intervals(3-30 min.) and the supernatants were filtered. The residual concentrations of Ni(II) ions were then determined by Atomic Absorption Spectrophotometer (Perkin Elmer AA Analyst 100 with air-acetylene oxidizing flame).

Figure 1: *Azaridchta indica* nut shells

Figure 2: NAINSC

3. Results and Discussion

3.1 Characterization by AFM &FTIR

Atomic Force Microscopic Analysis offers visualization and analysisin three dimensions. Using the AFM, individual particles and groups of particles can be resolved finer, compared to other microscopic techniques. Thus synthesised Nanohydroxyapatite-*Azaridachta indica* nut shell composite (NAINSC) was analyzed for the confirmation of its nano size, employing the AFM technique. The histogram of NAINSC (fig.3) shows a clear peak <18 nm which confirms the prevalent existence of maximum number of nanoparticles in the composite material. The three dimensional surface topography of NAINSC is also represented in figure 4.The FTIR spectrum of NAINSC is presented in figure 5

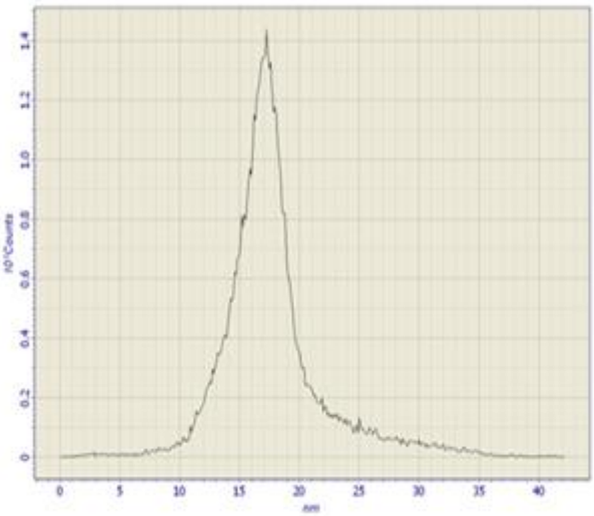

Figure 3: Histogram (NAINSC)

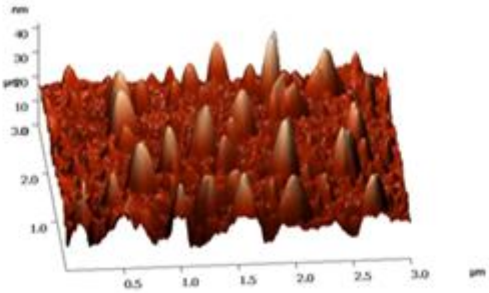

Figure 4: 3-D surface Topography (NAINSC)

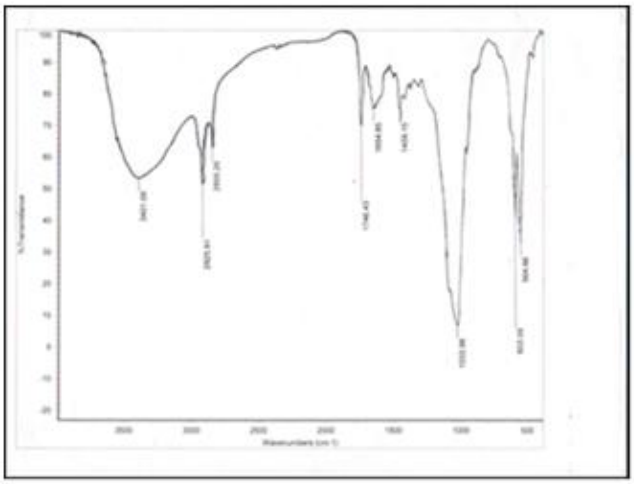

Figure 5: FTIR Spectrum of NAINSC

3.2. Effect of NAINSC dosage on Ni(II) removal

The efficiency of the synthesized NAINSC on Ni(II) was experimented by varying the composite dose(10-50 mg:10 mg interval). 11 ppm nickel solution was prepared from a stock of Ni(II) solution of 1000 mg/L was prepared by dissolving 6.7303 g of anhydrous Nickel ammonium sulphate in 1000 ml of double distilled water.Batch adsorption studies for different composite dosages with a contact time of 21 minutes for both the metal ions [Ni(II):pH 6.9; at 11 ppm initial metal ion concentration] were studied by varying the NAINSC dosage from 10 mg to 50mg. After agitation, the supernatant was filtered and analyzed using AAS.

The percentage removal of metal ions are presented in figure 7.15, the corresponding values are tabulated in table 7.4. The results show that 50 mg of NAINSC was sufficient to trap the divalent Nickel ion, where 74% of the metal has been removed, The sorption affinity of the metal on NAINSC was found to be good. This may be due to the solution pH, which is an important parameter in controlling composite sorption onto themetals. The physiochemical properties of n-HAP such as solubility, stability, crystallinity etc., vary depending upon the pH conditions[291]. At nearly a neutral pH of 6.9 employed for Ni(II)-NAINSC system, the composite material is probablyrelated to its lower solubility, This solubility nature of nano composites at neutral pH range enhances the availability of sorption sites, thereby increasing the percentage removal. Thus, the higher capability of NAINSC to adsorbNi(II), may be attributed to optimum pH conditions.

3.3 Effect of NAINSC dosage on industrial effluents

The eficiency of NAINSC was tested on industrial effluents collected from chemical and electroplating industries. The percentage removal of nickel from the respective effluents was observed to increase with dosages. Table -1support the experimental values of the NAINSC- Ni(II) systemto exhibit better nickel removal capacity.

Table 1: Effect of NAINSC dosage on Industrial effluent

Weight of adsorbent (mg)	% Removal of metal ions from effluents
	Nickel
10	69.61
20	70.58
30	70.62
40	70.76
50	71.18

4. Conclusion

From the above reports, it is concluded that the low-cost NAINSC was effective, easily available, non-toxic, indigenous adsorbents for the removal of Ni(II)from aqueous solutions and industrial effluents. The results of the present investigations would be useful for the fabrication and designing of wastewater treatment plants, utilizing such novel nanocomposites in an effective manner.Our future study aims at the establishment of performance oriented approach of NAINSC for other toxic heavy metal ions. This can be accomplished by developing suitable modification procedures in the sorbents viz. encapsulation and immobilization, thereby ensuring the safe disposal of the metal loaded sorbents, which in turn does not pose a great threat to the environment. Also the further work accomplishes to synthesize novel nanocomposites utilizing various agricultural waste materials to enhance and upgrade the sorption quality of such low cost sorbents.

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