

Adsorption of Ni (II) ions from aqueous solution Using Sulphuric Acid activated *Annona squamosa*



Chemistry

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ABSTRACT

The objective of this study is to assess the adsorption potential of Nickel removal from the aqueous solution by chemically activated Annona squamosa seed. The effects of various parameters such as solution pH, adsorbent dose, adsorbate concentration, contact time were analyzed. The maximum Nickel adsorption was obtained at pH 5. The surface morphology and functional groups of the activated carbon were determined by using SEM and FTIR analysis. The Ni (II) adsorption equilibrium data were analyzed by Langmuir, Freundlich, Temkin and Dubinin – Radushkevich (D-R) isotherm models. Kinetic studies showed that second order equation successfully describes the adsorption process. Thus Annona squamosa have good potential as an adsorbent to remove Ni (II) ions from aqueous solution.

INTRODUCTION

Metals are very important for human health but more hazardous when it exceeds the permissible limit. Arsenic, copper, lead, mercury, nickel and zinc have more polluted the environment. Nickel is harmful effluents of electroplating industries, smelting, alloy manufacturing, mining, refining, in welding rods, as a catalyst in oil hydrogenation. Permissible limit of Ni (II) in drinking water is 0.5 mg/L it enters the human body through inhalation, causing coughing, pneumonia, nasal cavities, stomach and prostate. There are various methods of removal of Ni(II) from industrial waste water include filtration, chemical precipitation, adsorption, and membrane systems or even ion exchange process. Among all these methods adsorption is one of the most economically favorable and a technically easy method. The paper deals with the investigation of Ni (II) removal from stimulated waste water by adsorption onto activated carbon (HAS) ¹.

EXPERIMENTAL PROCEDURE

Preparation adsorbent and adsorbate: *Annona squamosa* seed was collected from an agriculture farm in Andrapradesh. Then it was washed, dried and impregnated with sulfuric acid for 20 hrs and washed thoroughly with distilled water till it attained neutral pH and soaked in 2% NaHCO₃ over night in order to remove any excess of acid present and kept in a hot air oven at 300°C for carbonization. Then the material was named as HAS and preserved in air tight container for further use. 1000mg/L of Nickel sulphate hexahydrate was prepared as stock solution. Different concentrations of metal solutions were prepared by dissolving required amount of stock solution.

Methods and measurements: Batch adsorption experiments were carried out in a rotary shaker at 150 rpm using 250mL-shaking flasks with 0.2g HAS at room temperature for 120 min at pH 5.0. After shaking the flasks for 120 min, the mixture was analyzed for the residual Ni (II) ions concentration. The initial pH values of the solutions were previous adjusted with 0.1M HCl or NaOH using a DEEP VERSION model (EI) pH meter. The concentration of Ni (II) ions in solution was measured by UV visible spectrophotometer ².

RESULTS AND DISCUSSION

Characterization

SEM and EDAX analysis: The SEM and EDAX images clearly reveal the surface texture and morphology of the adsorbent is presented in Fig.1. The volatile matter was developed at high pressure, which emerge the cellular structure of the particle and created cavities on the surfaces of the carbon samples. During impregnation, the molecules of the chemical impregnating agent diffused into the adsorbent³.

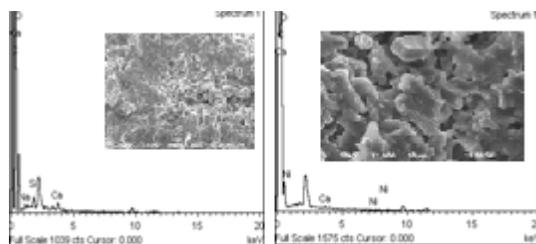


Fig.01: SEM and EDAX image of HAS with unloaded and loaded Ni (II)

Elemental composition of the adsorbent before and after was confirmed by its EDAX spectra. The EDAX spectrum showed the presence of C, O, Ca, Si, and Na peaks as component elements of the HAS sample. Among all these carbon and oxygen were the only common components in the HAS

FTIR analysis: Nickel loaded and unloaded HAS sample was analyzed in FTIR the region 500 cm⁻¹–4500 cm⁻¹. The peaks in FTIR spectrum of HAS indicates the presence of alkane C- H stretching,

N-H stretching, alkene C-H bending, and C-Cl stretching. It is clear that adsorbent displays a number of adsorption peaks, reflecting complex nature of adsorbent⁴.

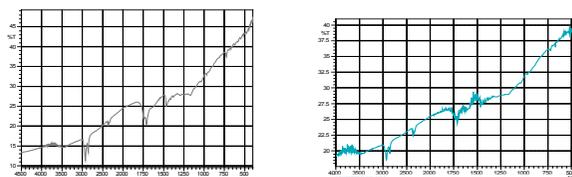
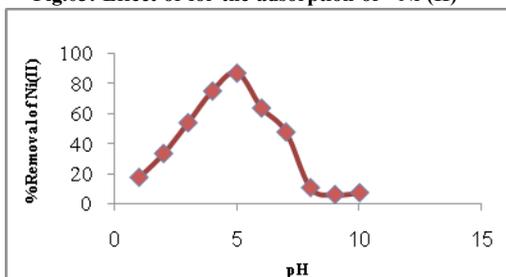


Fig.02: FTIR spectrum of Ni (II) loaded and unloaded HAS BATCH ADSORPTION STUDIES

Effect of pH: The pH of the system exerted a profound influence on the adsorptive uptake of adsorbate molecules, influence on the surface properties of the adsorbent and ionization of the adsorbate molecule. The maximum removal of Ni (II) was observed at pH 5. The pH at the zero point of charge (pH_{zpc}) of the HAS is reported to be 3.65. At lower pH the surface charge may get positively charged, thus (H⁺) ions compete effectively with metal cations causing a decrease in the amount metal adsorbed⁵.

Fig.03: Effect of for the adsorption of Ni (II)



pH onto HAS

Effect of Adsorbent dosage: The adsorption of Ni (II) on HAS was studied by changing the quantity of adsorbent 0.2, 0.4, 0.6, 0.8g and 1g in the test solution while keeping the initial concentration 50 mg L⁻¹. The increase in the Ni (II) ion adsorption with adsorbent dose increased from 0.2g to 1g. When adsorbent

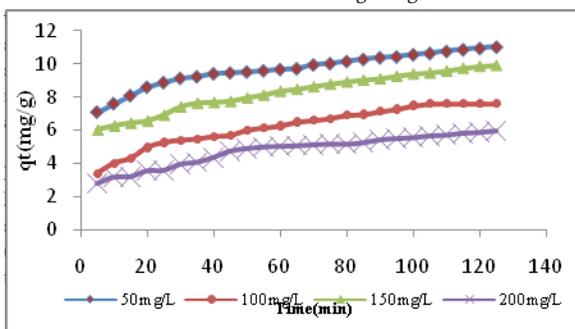


Fig.04: Effect of initial concentration and contact time for the adsorption of Ni (II) onto HAS.

Equilibrium Isotherms: Equilibrium data commonly known as adsorption isotherm, basic requirements for adsorption systems. In these studies Langmuir, Freundlich, Temkin and D-R isotherm models were analyzed. The isotherms for the adsorption of Ni(II) ions onto HAS with experimental conditions such as pH 5.0, contact time 120 min ,adsorbent dose 0.2g/50mL are shown in fig 2 and 3. All isotherm parameters and values presented in Table.1. From Langmuir equation adsorption capacity Q_m was 34.012mg/g. The Freundlich constants K_f and n values are 1.027 and 2.855 respectively. n values between 0 and 10 indicates a favorable adsorption of Ni (II) onto HAS. A comparative data from Table.1 Langmuir and Temkin models showed better fit compared to Freundlich and Dubinin-Radushkevich models⁸.

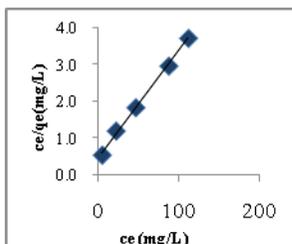
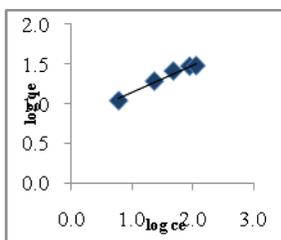


Fig.05:Langmuir and Freundlich isotherms for Ni(II) adsorption onto HAS

Table.1 Isotherm and kinetic parameter for the adsorption of Ni(II) onto HAS

Isotherm Models	
1.Langmuir	
Q_m (mg g ⁻¹)	34.012
K_a (L mg ⁻¹)	0.0128
R^2	0.9979
2. Freundlich	
$1/n$ (g l ⁻¹)	2.855
K_f (mg g ⁻¹)	1.027
R^2	0.9794
3. Temkin	
α (L/mg)	5.0296
β (mg/L)	0.1473
R^2	0.9922
4. Dubinin-Radushkevich (D-R)	
Q_m (mg g ⁻¹)	176.18
K (x 10 ⁻⁵ molKJ ⁻²)	2.4154
E (KJ/mol)	0.4550
R^2	0.9508
Kinetic models	
1.Second-order kinetic model	
K_2 (mg min ⁻¹)	0.066
q_c (mg g ⁻¹)	11.36
R^2	0.9965
Experimental value	
q_c (mg g ⁻¹)	11.32
2. Elovich	
A_p	1.2505
B_p	23.52
R^2	0.9813
3. Intra particle Diffusion	
K_{diff}	2.451
C	16.059
R^2	0.9732

Adsorption Kinetics: The Kinetic parameters, which are helpful to find out adsorption rate, give important information for modeling the adsorption processes. The kinetics of Ni (II) onto HAS were analyzed using pseudo-first-order, pseudo-second-order, intraparticle diffusion and Elovich kinetic models. The experimental adsorption capacity (q_{exp}) 11.32 is very close to calculated (q_{cal}) 11.36 from Second-order Kinetic model. Which led to believe that pseudo-second-order Kinetic model provided good correlation for the adsorption of Ni (II) and onto HAS⁹.

CONCLUSION

The results revealed the activated carbon developed from *Annona squamosa* has a suitable adsorption capacity for the removal of Ni (II) ions from aqueous solutions. Comparing other isotherms Langmuir and Temkin are well fitted¹⁰. The data indicate that the Adsorption kinetics followed a pseudo-second-order rate with intraparticle diffusion as one of the rate determining steps. HAS can be employed as low-cost adsorbents as alternatives to commercial activated carbon for the removal of Ni (II) from water and wastewater.

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