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Analytical Instrumentation Techniques of FT-IR, XRD, SEM, and EDX for Adsorption Methods of Ni^{2+} Ions onto Low Cost Adsorbent

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ABSTRACT

In present investigate the possible removal of Ni^{2+} ions from aqueous solution by using low-cost *Hygrophila auriculata* activated nano carbon (HA-ANC) as an adsorbent. The activated nano carbon had been prepared from *Hygrophila auriculata* stem waste as well; the raw material was carbonized with con. H_2SO_4 and activated by thermal action. Batch experiments were performed in order to calculate the percentage removal of Ni^{2+} ions for 90.737% at 60°C. The properties of treated carbon and untreated carbon are compared using instrumental techniques such as FT-IR, XRD, SEM and EDX, which confirms Ni^{2+} ions adsorption onto HA-ANC. FT-IR showed that the surface of HA-ANC had more oxygen containing functional groups which enhanced the adsorption of Ni^{2+}. XRD showed the nature of adsorbent, SEM images implies morphological deviance of before and after adsorption of Ni^{2+} onto HA-ANC and EDX showed that the C content of HA-ANC were higher than that of Ni^{2+}/HA-ANC.

Keywords: Adsorption, Hygrophila auriculata activated nano carbon (HA-ANC), Batch method, Ni^{2+} ions, FT-IR, XRD, SEM, EDX.

INTRODUCTION

Pollution of heavy metals has attracted global attention due to their toxicity, hard decay and accumulation in organisms. Heavy metals accumulate in the tissues of various living organisms and as a result attach to the food chain, affecting humans, posing a health risk[1-3]. Therefore, the treatment of wastewater polluted by heavy metals is an important environmental concern. Ion exchange, solvent extraction, chemical precipitation, ultra-filtration, reverse osmosis, electro dialysis and adsorption are the traditional methods for removing heavy metal ions from polluted water[4-6]. However, absorption is considered to be one of the most popular methods for removing heavy metals from wastewater due to its low cost, in effect, biodegradability, ease of design and high removal efficiency. There are many absorbent materials available, but activated carbon is used to remove heavy metals by the adsorption method because activated carbon has a numerous pores and a large surface area[7,8].

The literary study shows that no work has been done on *Hygrophila auriculata* activated nano carbon (HA-ANC) as an adsorbent, as well as low cost and high abundance. Therefore this study focused on the removal of Ni^{2+} ions from the aqueous solution using the HA-ANC by batch adsorption method.

MATERIALS AND METHODS

Chemicals

Analytical reagent grade chemicals were used. Stock Ni^{2+} ion solution (1000ppm) was prepared by dissolving required amount of $NiSO_4.6H_2O$ in 1000 mL of double distilled water. Working standards were prepared by diluting the stock solution of Ni^{2+} ions using double distilled water. The ionic solutions of 0.1M HCl and 0.1MNaOH were made for to alter the solution pH.

Procedure for adsorbent preparation and activation

The *Hygrophila auriculata* stem waste was obtained from the agricultural sites near at Poompuhar, Mayiladuthurai district, Tamilnadu, India. The stem was cut into small pieces, dried in the absence of sunlight and treated with concentrated H_2SO_4 in W/V ratio. The carbonized material washed away by double distilled water until it becomes neutral one and dried further inside the hot air oven at 110°C for 24 hours. The carbon was activated by using muffle furnace at 1000°C for 6 h and the activated carbon (HA-ANC) was powdered well and stored in desiccators in order to perform the experiment[9].

Batch adsorption method

The batch experiments[10] for Ni^{2+} ions removal was determined under various initial pH like 3, 4, 5, 6, 7, 8 and 9 besides temperature ranges from 30 to 60°C at an initial Ni^{2+} ion solution concentration was varied from 10 to 50ppm with different adsorbent doses and different time intervals such as 15, 30, 45, and 60 minutes. The effect of initial Ni^{2+} ions concentrations was investigated by using 25 mg biomass, initial pH 6 and volume of Ni^{2+} ion solution 50 mL are constant but varied initial Ni^{2+} concentrations at room temperature (30°C). The Ni^{2+} ion solution samples were collected after 60 min of shaking then centrifuged at 120rpm, the filtrate was analyzed. The unadsorbed Ni^{2+} ions were measured by using UV-Visible spectrophotometer. The removal percentage and amount of Ni^{2+} ions adsorption were calculated using equation 1 and equation 2 correspondingly.

Mass balance	Relationship equation	Eq. No.
Removal percentage	$\% R = \left(\frac{C_0 - C_e}{C_0}\right) \times 100$	(1)
Amount of adsorption	$q_e = v \times \frac{(C_0 - C_e)}{w}$	(2)

Where, %R is the percentage of removal, q_e is the amount of adsorbed Ni^{2+} ions per unit quantity of adsorbent at equilibrium time (mg g^{-1}), C_0 and C_e are the initial and actual concentration (ppm) of Ni^{2+} ion solutions at equilibrium time, respectively. The V is the volume of the Ni^{2+} ion solution (L), 'w' is the weight of the HA-ANC (g).

Analytical tools for HA-ANC and Ni^{2+}/HA-ANC

The determination of surface area, volume and diameter of pores for HA-ANC, BET-BJH methods were used. The FT-IR spectrum is an important tool to study the changes infrequency due to the interaction between adsorbent and adsorbate. The XRD pattern confirms the nature of the adsorbent as well as adsorption of Ni^{2+} ions onto HA-ANC in order to changes of 2θ values. SEM is a substantial tool to evaluate the morphological features and surface characteristics of the fresh adsorbent and treated adsorbent materials and also EDX study gives details about the element compositions[11,12].

RESULTS AND DISCUSSIONS

The equilibrium data was calculated with the help of batch methodology it's incorporated some of the effective parameters such as contact time, solution pH, adsorbent dose, temperature[13]. The data was given in Table 1, the result says that the nature of effectiveness of Hygrophila auriculata activated nano carbon (HA-ANC), which indicates the amount of adsorbed Ni^{2+} ions onto HA-ANC increased with increasing temperature. The removal was high (90.737%) at 60°C it's also declared the removal of Ni^{2+} ions onto adsorbent favored in elevation of temperature as well as low concentration of Ni^{2+} ion solution by 25 mg of adsorbent.

Table 1: Equilibrium Data for the Adsorption of Ni2+ ions onto HA-ANC

C_0	C_e(Mg / L)				q_e(Mg / L)				Removal%			
	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C
10	1.804	1.463	0.976	0.926	16.393	17.075	18.048	18.147	81.963	85.375	90.238	90.737
20	5.363	4.877	4.408	3.414	29.275	30.246	31.184	33.173	73.188	75.614	77.960	82.932
30	11.149	10.630	9.263	8.582	37.702	38.741	41.475	42.836	62.836	64.568	69.125	71.393
40	17.103	15.641	14.837	13.917	45.793	48.718	50.327	52.166	57.241	60.897	62.908	65.208
50	25.350	24.471	23.931	22.510	49.300	51.059	52.139	54.981	49.300	51.059	52.139	54.981

According to the BET-BJH method, the surface area, volume and diameter of pores was obtained as 90.067 m²/g, 0.115 cc/g and 3.520nm, respectively.

Fourier transform infrared was used to determine the changes of vibration frequency in the functional groups of the adsorbent due to Ni^{2+} ions adsorption. The FT-IR spectrum within 500-4000 cm^{-1} for the HA-ANC before and after the adsorption of Ni^{2+} is shown in order to Fig. 1 and Fig. 2. The peak point of frequency; 2500 to 4000 cm^{-1} indicates the single bonds (C-H, O-H, N-H), 2000 to 2500 cm^{-1} indicates the triple bonds (C≡C, C≡N), 1500 to 2000 cm^{-1} indicates the double bonds (C=C, C=N, C=O) and 500 to 1500 cm^{-1} indicates fingerprint region. The FT-IR spectrum of HA-ANC indicates that there is remarkable change in the peaks at 2997.49 cm^{-1},2888.47 cm^{-1}, 1860.25 cm^{-1}, 1560.72 cm^{-1}, 1184.19 cm^{-1}, 1123.42 cm^{-1}, 1035.85 cm^{-1}, 795.09 cm^{-1}, and 525.96 cm^{-1} respectively which it can be due to Ni^{2+} binding with functional groups adsorbent. The formation of new peaks, demolition of old peaks and higher to lower as well as lower to higher shifting of peaks were reason for that HA-ANC adsorbed with Ni^{2+} ions[14-16].

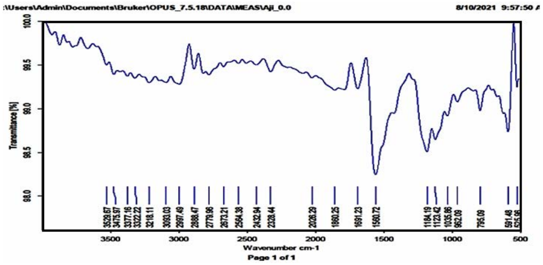

Fig. 1. FT-IR for HA-ANC

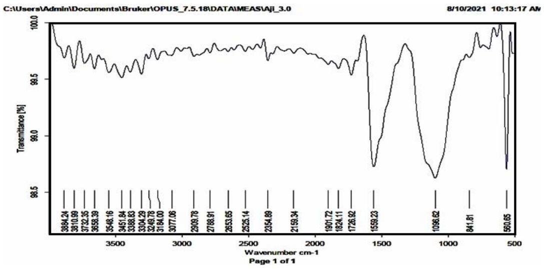

Fig. 2. FT-IR for Ni^{2+}/HA-ANC

The result of XRD diffractogram for HA-ANC (Fig. 3) revealed that the *Hygrophila auriculata* activated nano carbon is crystalline in nature and resembles the graphite structure[17-19]. After adsorption of Ni^{2+} ions (Fig. 4) the surface of adsorbent was disturbed this one leads to new theta values, HA-ANC given in Table 2 and Ni^{2+}/HA-ANC given in Table 3 respectively.

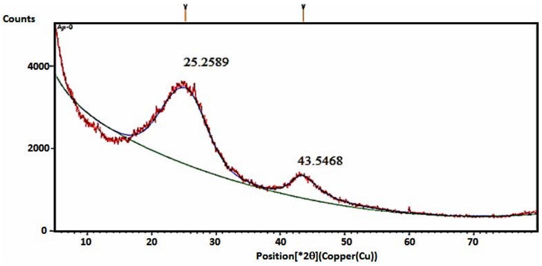

Fig. 3. XRD for HA-ANC

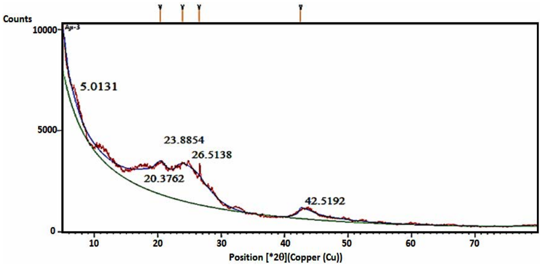

Fig. 4. XRD for Ni^{2+}/ HA-ANC

Table 2: XRD measurements of HA-ANC

Pos.[°2θ]	Height[cts]	FWHM Left[°2θ]	d-spacing[Å]	Rel.Int.[%]
25.2589	1233.20	8.7438	3.52305	100.00
43.5468	384.92	4.5991	2.07663	31.21

Table 3: XRD measurements of Ni^{2+}/HA-ANC

Pos.[°2θ]	Height[cts]	FWHM Left[°2θ]	d-spacing[Å]	Rel.Int.[%]
5.0131	23772.90	0.0520	17.61333	100.00
20.3762	708.42	3.3002	4.35492	2.98
23.8854	1206.41	5.9034	3.72244	5.07
26.5138	209.24	9.0362	3.35909	0.88
42.5192	718.56	0.9656	2.12441	3.02

SEM images have been used for morphological study of HA-ANC. The SEM micrographs of HA-ANC before and after adsorption of Ni^{2+} ions are shown in Fig. 5 and Fig. 6 respectively. In the SEM image of HA-ANC, the porous structure is obvious, but in the case of the used particles, the pores have been covered by the Ni^{2+} ions[20,21].

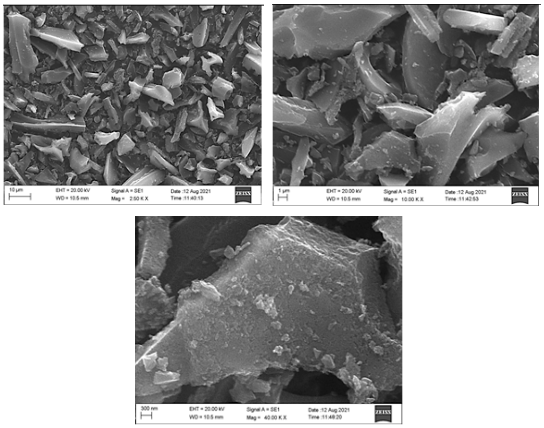

Fig. 5. SEM image for HA-ANC in different magnifications 10 μm, 1 μm and 300 nm

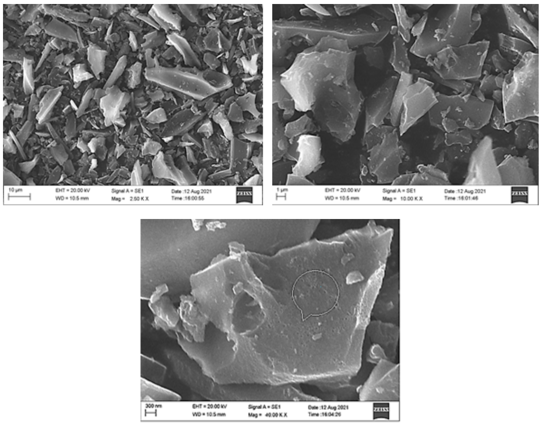

Fig. 6. SEM image for Ni^{2+}/ HA-ANC in different magnifications 10 μm, 1 μm and 300 nm

The EDX spectrum[22,23] of HA-ANC before and after adsorption of Ni^{2+} ions were evaluated shown in Fig. 7 and Fig. 8 respectively. The spectrum clearly indicates the peak of element C, there was significant reduction in the intensity of peak of C in the after adsorption of Ni^{2+} ions comparing with the before adsorption. And also the peak of Ni^{2+} ions was notable on the after adsorption of Ni^{2+} ions onto HA-ANC in addition the elemental compositions statistics were given in Table 4 and Table 5 respectively for before and after adsorption Ni^{2+} ions onto HA-ANC. In addition the atomic percentage of carbon reduced form 99.7% to 88.8% bassed on Table 4 and Table 5, it can be usable as remarkable reduction.

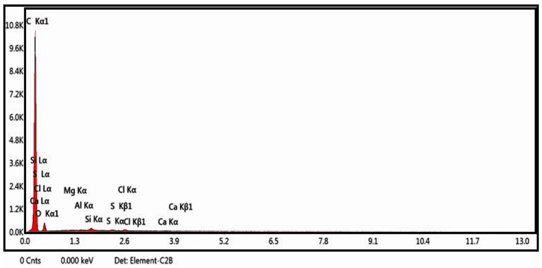

Fig. 7. EDX for HA-ANC

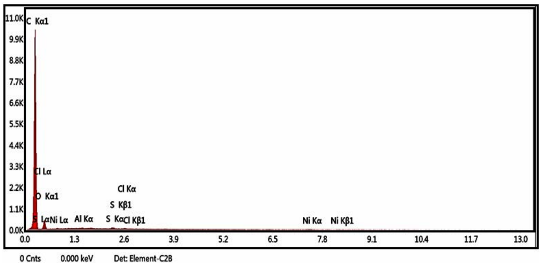

Fig. 8. EDX for Ni^{2+}/HA-ANC

Table 4: EDX of Smart Quant Results for HA-ANC

Element	Weight%	Atomic%	Error%	Kratio
CK	85.6	89.7	4.4	0.5959
OK	11.7	9.2	13.9	0.0133
MgK	0.5	0.2	13.2	0.0029
AlK	0.4	0.2	13.2	0.0026
SiK	0.7	0.3	7.2	0.0056
SK	0.3	0.1	18.3	0.0028
ClK	0.4	0.1	19.9	0.0035
CaK	0.4	0.1	27.7	0.0034

Table 5: EDX of Smart Quant Results for Ni^{2+}/HA-ANC

Element	Weight%	Atomic%	Error%	Kratio
CK	84.8	88.8	4.0	0.6254
OK	13.5	10.6	13.6	0.0158
AlK	0.4	0.2	9.9	0.0031
SK	0.4	0.2	15.4	0.0038
ClK	0.2	0.1	30.2	0.0020
NiK	0.6	0.1	20.0	0.0062

CONCLUSION

The *Hygrophila auriculata* activated nano carbon (HA-ANC) has been used successfully with an initial concentration of 10ppm at pH 6 in 60 minutes. The appearance, disappearance and shifting of peaks in the FT-IR spectrum confirmed the adsorption of Ni^{2+} ions onto HA-ANC as well as XRD analysis also provided reasonable evidence for adsorption to the adsorbent. In addition to supporting, the SEM study gives difference in the surface morphology of the adsorbent before and after the adsorption of Ni^{2+} ions as well as EDX also given elemental composition evident of removal of Ni^{2+} ions onto HA-ANC. Finally, it was concluded that the present adsorbent HA-ANC could be a good alternative adsorbent for the removal of heavy metals from aqueous solutions in a very efficient and economical manner.

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Conflict of interest

The author declare that we have no conflict of interest.

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