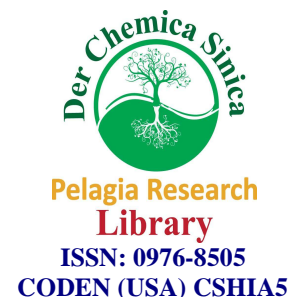




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Studies on the removal of nickel(II) using chemically activated pouteria sapota seed and commercially available carbon

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ABSTRACT

The capacity of sulphuric acid treated Pouteria sapota seeds carbon for the removal of Nickel from aqueous solution was examined. The influence of various parameters such as effect of equilibration time, pH, and carbon dosage for the removal of Nickel was studied using batch process. The experiment demonstrated showed that equilibrium adsorption data fit the Freundlich adsorption isotherm. The adsorption behavior of the activated carbon is explained on the basis of its chemical nature. Desorption study was carried out using HCl solution with a view to regenerate the spent adsorbent and to recover the adsorbed metal ion.

Keywords: pouteria sapota seed, Freundlich adsorption isotherms.

INTRODUCTION

Nickel is shown to be essential for some microorganisms and animals but not to plants. It is associated with the synthesis of vitamins. It is toxic at higher concentration. Nickel and its salt are used in several industrial applications such as in electroplating, storage batteries and automobile and aircraft parts, printing fabrics. However Nickel is found to be toxic for most plants and fungi. Growth of the plants will be affected when the concentration of nickel in the soil is high. It causes cancer, depression, heart attacks, hemorrhage, kidney dysfunction, low blood pressure, muscle tremors, paralysis, nausea, tetanus and chest pain[1]

The conventional methods for the removal of Nickel from waste water are supplied precipitation[2,3], coagulation and ion exchange procedures. But due to operational difficulties, these methods have limited applications. Adsorbents such as flyash[4], algal biomass [5]low rank coal[6], certain clay and clay minerals[7], have been used but with limited success. The attention has shifted to activated carbon adsorbents which have large surface area and a high degree of surface reactivity.

OBOH et al.,[8] I studied the bioadsorption of Nickel(II) ions from aqueous solutions using a biomaterial . Mohammad Shakirullah etal., found out the sorption studies of nickel ions onto sawdust of Dalbergia sissoo[9]. Biosorptive removal of nickel from wastewater and industrial effluent was investigated by Piyush Kant Pandey et.al,[10].Chemical modification of oil palmfruit fibre by ABIA, et.al[11]increased the percentage removal of Ni²⁺.Orange peel cellulose adsorbents was investigated by Fang Luoa et.al, [12]for the removal of nickel ions. Removal of trace-level nickel from surface water using different nickel sorbents in column were carried out by Kllassor, K. ,J. [13].

Development of new economically feasible eco-friendly products from nature for removal of pollutants from aqueous solution is the objective of our continued research. The present study is undertaken with a view to assess the feasibility of acid treated pouteria sapota seed shell carbon (SPSSC) as an adsorbent for the removal of Nickel(II). The effects of various parameters such as equilibration time, adsorbent dose, pH, Freundlich adsorption isotherm and desorption studies on the adsorption process have been studied.

MATERIALS AND METHODS

Preparation of adsorbent

Preparation of sulphuric acid treated pouteria sapota seed shell carbon(SPSSC)

Several 50g lots of pouteria sapota seed shell powder were carbonized using 20ml of concentrated sulphuric acid, to get sufficient quantity of carbon for systematic studies of nickel removal. After mixing thoroughly, the samples were let stand in an air oven at 140-160°C for 24hrs to facilitate charring of the material. They were then washed free from acid using tap water and finally with distilled water. The washing solutions were discarded and the carbon was dried in an air oven at 110°C.

Particles are separated by passing it through 50 mesh and above which constituted 20% of the bulk material were rejected. Particles in the size ranges 20-50 mesh are retained for detailed study. Particles in the size greater than 20 mesh were carefully ground and sieved to the desired range(20-50) mesh were preserved for detailed experimental studies.

Similarly, commercially available carbon was also dried, powdered and sieved to (20-50) mesh which is used as another adsorbent(CAC)

Preparation of Nickel Stock solution:

About 447.9mg of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ is dissolved in distilled water and made upto 1000mL in a volumetric flask.

Batch Experiments

Batch mode adsorption studies were carried out by agitating 100ml of Nickel(II) solution at desired concentration with different dose of adsorbent. The samples were filtered and finally 20ml of 0.5N HCl, 10ml of sodium citrate, 2ml of iodine solution and 4ml of dimethyl glyoxime were added to the filtrate and analyzed by spectrophotometer at 575nm for the estimation of Nickel(II). Effects of equilibration time, adsorbent dosage, pH were studied. Freundlich adsorption and desorption studies were also carried out.

RESULT AND DISCUSSION

Characteristics Of The Adsorbents[14,15]

The characteristics of SPSSC and CAC are presented in Table 1. It was observed that the sulphuric acid treated Pouteria sapota seed shell carbon and commercially available carbon(CAC) have bulk density of 0.7181 and 0.1977g/cc respectively. The moisture content of SPSSC and CAC was found to be 4.3580% and 5.559% respectively. The ash content was estimated to be 25.5480% and 3.0283% respectively whereas the iron content was found to be 50% and 75% respectively. The matter soluble in water and in acid for SPSSC and CAC was found to be 1.5, 0.2181 and 4.3, 1.6433 respectively. The decolourising power of SPSSC was 21mg/g whereas for CAC was 480mg/g. SPSSC has pH equal to 5.23 while CAC has a pH of 1.6433

Effect Of Equilibration Time

Effect of contact time was studied by taking 10ppm nickel solution at pH5 with an adsorbent dose of (0.5g/100ml). Rate of uptake of Nickel(II) was initially higher and reached a steady value after reaching equilibration at 3rd and 4th hour for SPSSC and CAC. The removal percentage was found to be 80 for SPSSC and 74 for CAC. The increase in removal efficiency with contact time is due to the increase in surface area and hence more active sites are available for adsorption[16].

Effect Of pH

The influence of pH of the synthetic effluent on the extent of adsorption of Nickel(II) is shown in Table 3 and fig 2. The percentage of adsorption of the metal increases with increase in pH. Maximum removal was observed at pH

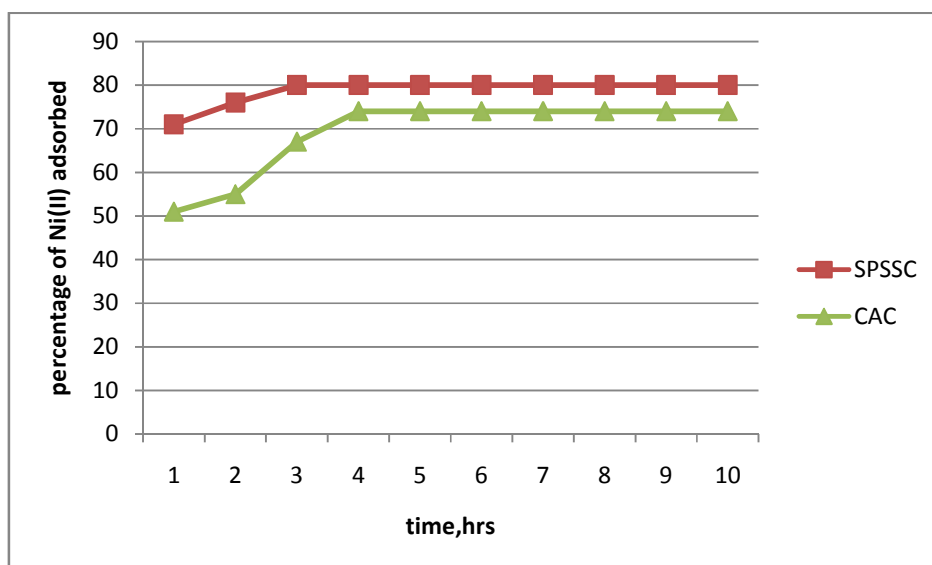
3.0-10.0 for SPSSC and at pH 5.0-10.0 for CAC. At higher pH the reduction in adsorption may be possibly due to the abundance of OH⁻ ions creating increased hinderance to the diffusion of nickel ions [17].

Table 1. Characteristics of Carbon

Characteristics	Acid treated pouteria sapota seed shell carbon(SPSSC)	Commercially Available Carbon(CAC)
Moisture Content	4.3580	5.559
Ash Content	25.5480	3.0283
Apparent Density (or) Bulk Density g/cc	0.7181	0.1977
Matter soluble in water	1.5	0.2181
Matter soluble in acid	4.3	1.6433
pH	5.23	6.79
Decolourizing power (mg/g)	21	480
Iron Content (%)	50	75
Ion exchange capacity	0.06	0.5

Table 2. Effect of Equilibration time

Time, hr	Percentage of Nickel(II) adsorbed	
	SPSSC	CAC
1	71	51
2	76	55
3	80	67
4	80	74
5	80	74
6	80	74
7	80	74
8	80	74
9	80	74
10	80	74

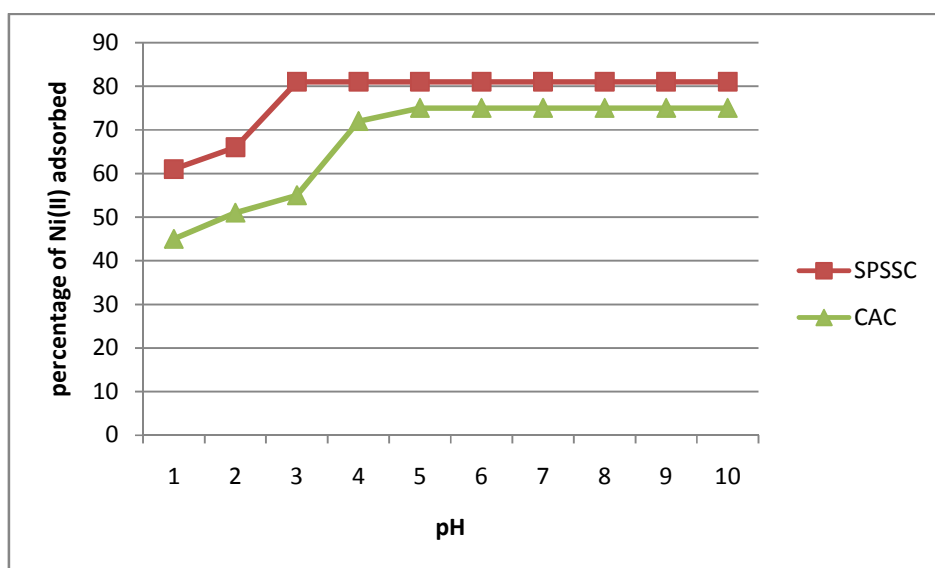
**Figure 1. Effect of Equilibration time**

Effect Of Carbon Dose

The effect of adsorbent dosage level on sorption of nickel is shown in Table 4 and fig 3. Study was carried out by taking 5ppm nickel solution. Adsorbent dosage varied from 50mg/L to 500mg/L. 80% and 75% removal occurred at a dosage level of 100mg/L and 150mg/L for SPSSC and CAC respectively. The increase in adsorption with increase in adsorbent dose is due to increase in the surface area of the adsorbents and hence more active sites are available for the adsorption of the metal ion [18]

Table 3. Effect of pH

pH	Percentage of Nickel(II) adsorbed	
	SPSSC	CAC
1	61	45
2	66	51
3	81	55
4	81	72
5	81	75
6	81	75
7	81	75
8	81	75
9	81	75
10	81	75

**Figure 2. Effect of pH****Table 4. Effect of Carbon dosage**

Carbon dosage mg/100mL	Percentage of Nickel(II) adsorbed	
	SPSSC	CAC
50	74	55
100	80	65
150	80	75
200	80	75
250	80	75
300	80	75
350	80	75
400	80	75
450	80	75
500	80	75

Freundlich Adsorption Isotherm

The adsorption isotherm indicates how the adsorbed molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state[16]

To explore the insights into the adsorption process the most common way is to analyse the adsorption isotherm which is normally obtained by plotting the adsorbed amounts versus the equilibrium concentration of adsorbate solution[17]. In the present study Freundlich isotherm has been considered.

The applicability of Freundlich isotherm was tried using the following general equation.

$$x/m = K_f C_e^{1/n}$$

Therefore $\log x/m = \log K_f + 1/n \log C_e$

K_f and n are constants representing the adsorptive capacity and intensity of adsorption respectively. Obviously a plot between $\log x/m$ and $\log C_e$ would be a straight line. Such a representative linear plot for SPSSC and CAC are shown in fig 4 and 5 and the corresponding data are presented in table 5, 6. The linearity obtained for both the carbons shows that there is favourable adsorption of Nickel(II) [87].

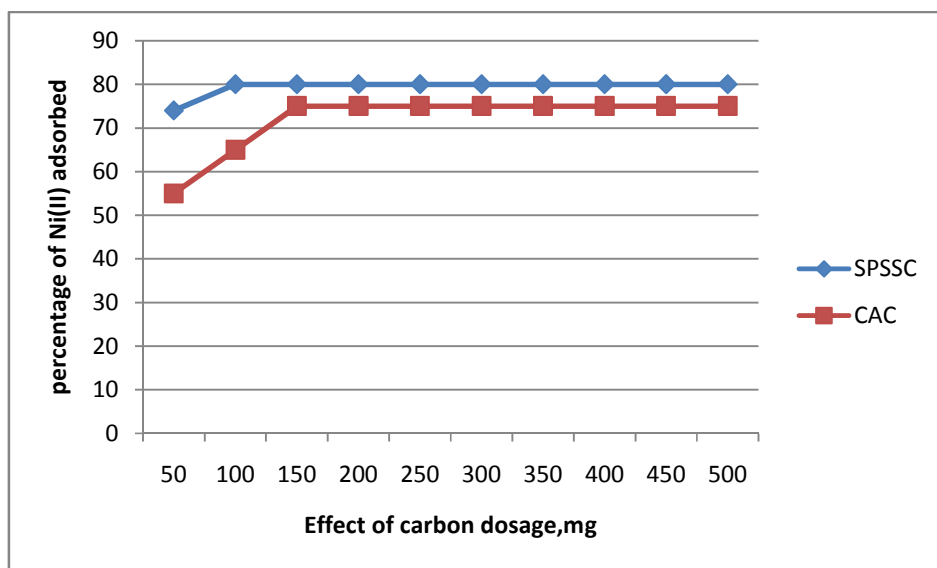


Figure 3. Effect of carbon dosage

Table 5. Freundlich adsorption isotherm for SPSSC

Concentration of solution, ppm	x/m	$\log x/m$	$\log C_{eq}$
10	7.3	0.8633	1.0000
20	14.8	1.1702	1.3010
30	22.5	1.3521	1.4771
40	30.4	1.4828	1.6020
50	38.4	1.5843	1.6989
60	46.1	1.6637	1.7781
70	53.6	1.7291	1.8451
80	61.1	1.7860	1.9031
90	69.1	1.8394	1.9542

Desorption Studies

Desorption studies help to elucidate the nature of adsorption and to recover the precious metals from waste water using the adsorbent[20]. Attempt was made to desorb the metal from spent carbons using hydrochloric acid of various strengths. In highly acidic medium, protons(H^+ ions) displaces the Nickel(II) ions by ion exchange mechanism. It was found that nickel desorption increased with increase in the concentration of the acid[21]. Table 7 showed that a concentration of 0.1N HCl was required to recover 79% of SPSSC and 74% CAC.

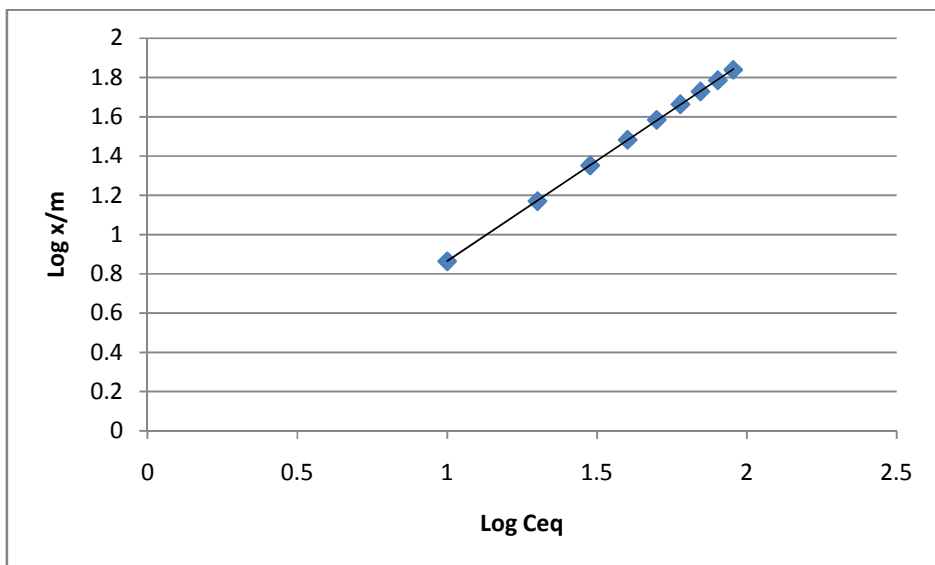


Figure 4. Freundlich Adsorption isotherm for SPSSC

Table 6. Freundlich adsorption isotherm for CAC

Concentration of solution, ppm	x/m	Log x/m	Log Ceq
10	3	0.4771	1.0000
20	5	0.6989	1.3010
30	15	1.1761	1.4771
40	26	1.4149	1.6020
50	33	1.5185	1.6989
60	42	1.6232	1.7781
70	48	1.6812	1.8451
80	59	1.7709	1.9031
90	70	1.8450	1.9542

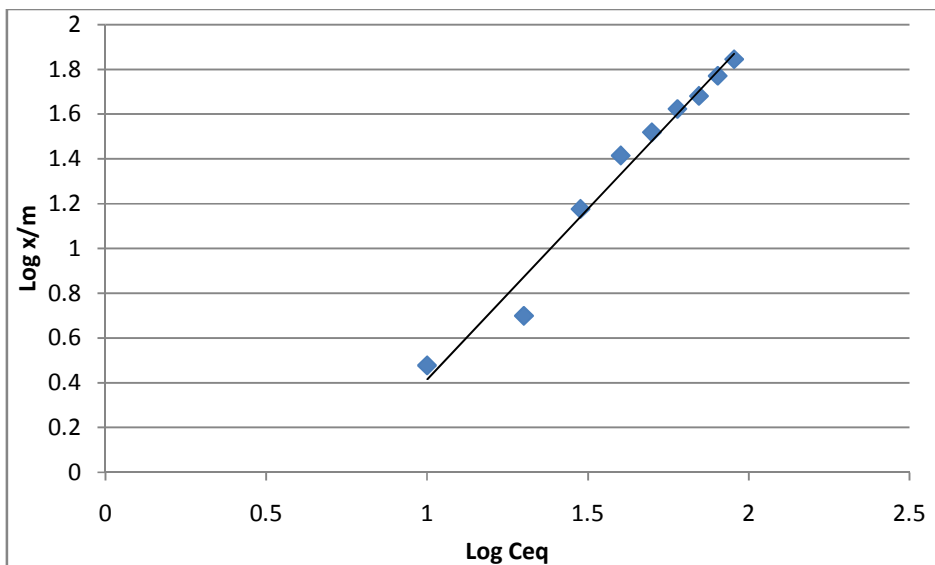


Figure 5. Freundlich Adsorption Isotherm for CAC

Table 7. Desorption studies

Normality of HCl(N)	Percentage of Nickel(II) adsorbed	
	SPSSC	CAC
0.01	24	25
0.02	29	30
0.03	36	40
0.04	41	45
0.05	49	52
0.06	57	57
0.07	61	62
0.08	68	67
0.09	75	71
0.10	79	74

CONCLUSION

1. Examination of carbon characteristics such as moisture content, ash content, matter soluble in acid, matter soluble in water, pH, decolorizing power, ion exchange capacity, apparent density and iron content showed the suitability of acid treated castor seed shell carbon(SPSSC) to be higher than commercially available carbon(CAC) for the removal of Nickel(II) ions.

2. By batch mode studies, the removal was found to increase with time and attains equilibrium at 3rd hour and 4th hour for SPSSC and CAC respectively with the removal percentage of 80 for SPSSC and 74 for CAC 3. The percentage removal reached a maximum at pH 3 for SPSSC and at pH 5 for CAC

4. The removal of Nickel(II) was taken as a function of carbon dosage. The carbon dosage obtained for SPSSC and CAC was 100mg/100mL and 150mg/100mL respectively.

5. Regeneration studies has been found that 0.01N of HCl is required to recover 79% of SPSSC and 74% of CAC

6.. Freundlich isotherm was found to be linear indicating the classical Applicability of the adsorption isotherm to the adsorbate- adsorbent system.

Hence it has been found that from the reported data SPSSC may be very useful for designing an economically cheap treatment process for the removal and recovery of Nickel(II) from nickel containing waste water in chemical and allied process industries than CAC.

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