

## Adsorption of Nickel (II) from Aqueous Solution by Bicarbonate Modified Coconut Oilcake Residue Carbon

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The adsorption of Ni (II) on modified coconut oilcake residue carbon (bicarbonate treated coconut oilcake residue carbon-BCORC) was employed for the removal of Ni (II) from water and wastewater. The influence of various factors such as agitation time, pH and carbon dosage on the adsorption capacity has been studied. Adsorption isothermal data could be interpreted by Langmuir and Freundlich equations. In order to understand the reaction mechanism, kinetic data has been studied using reversible first order rate equation. Similar studies were carried out using commercially available activated carbon – CAC, for comparison purposes. Column studies were conducted to obtain breakthrough capacities of BCORC and CAC. Common anions and cations affecting the removal of Ni (II) on both the carbons were also studied. Experiments were also done with wastewater containing Ni (II), to assess the potential of these carbons.

**Key words :** *Adsorption, Nickel (II), aqueous solution, bicarbonate, coconut oilcake residue, carbon, Langmuir and Freundlich equations*

### Introduction

Heavy metals are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders. Significant quantities of Nickel – containing wastewater are introduced into water bodies from the effluent of nickel-plating plants, silver refineries, zinc base casting industries and storage batteries<sup>1</sup>. The tolerance limit of nickel in drinking water is 0.01mg/L<sup>2</sup>. Higher concentration of nickel causes cancer of lungs, nose and bone. Acute Ni (II) poison causes dizziness, headache, nausea and vomiting, chest pain, dry cough and shortness of breath, rapid respiration, cyanosis and extreme weakness<sup>3</sup>. These harmful effects of Ni (II) necessitate its removal from wastewater before release into streams.

Many reports are available on the development of activated carbon from cheaper and readily available materials such as rice husk, coconut shell etc<sup>4,6</sup>. In this study, carbon from coconut oilcake residue, a waste agricultural byproduct obtained from oil industries, utilized for the removal of Ni (II) from water and wastewater. The investigation reported here deals with a comparative study of BCORC and CAC for the removal of Ni (II) from aqueous solution and from a Nickel-plating industry wastewater.

### Material and methods

#### *Preparation of carbons*

Coconut oilcake residue procured from oil industries was washed with distilled water, dried at 110°C, cut into small pieces and sieved to 0.575mm (20-50ASTM) mesh size. Then it was treated with concentrated sulphuric acid in 1:1 weight ratio and kept in an air-oven at 150±5°C for 24 hrs. The carbonized material was washed with distilled water to remove the free acid and dried at 105±5°C. Then it was repeatedly soaked in 2% sodium bicarbonate until effervescence ceased and further soaked in the same solution for two days to remove any residual acid. The material was then washed with distilled water, dried at 105±5°C and again sieved to 20-50 ASTM mesh size (BCORC). Preliminary studies were carried out with raw coconut oilcake residue, sulphuric acid treated coconut oilcake residue and bicarbonate treated coconut oilcake residue for the removal of Ni (II). Based upon their efficiency, bicarbonate treated CORC was chosen for further studies. The characteristics of carbon were found out using BCORC. The CAC (SD fine chemicals) was procured from the market and sieved to 20-50ASTM mesh size and characteristics were also

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**Table 1 :** Characteristics of BCORC and CAC carbons

S.No.	Description	Results	
		BCORC	CAC
1.	Bulk (or) apparent density (g.mL <sup>-1</sup> )	0.54	0.66
2.	Moisture (%)	6.78	13.4
3.	Ash (%)	5.5	2.4
4.	Matter Soluble in water (%)	1.93	1.15
5.	Matter Soluble in acid (%)	6.44	19.86
6.	pH	7.6	8.15
7.	Decolorizing power (MBT) (mg/g)	6.75	4.5
8.	Phenol Number	20.78	12.25
9.	Ion Exchange Capacity (m.equiv.g <sup>-1</sup> )	0.75	Nil
10.	Surface area (sq.m /g)	211	214
11.	Iron (%)	0.73	1.29

found out. The characteristics of BCORC and CAC are summarized in **Table 1**.

### Batch experiments

A stock solution of 100mg/L of Ni (II) was prepared. Batch experiments were conducted in Polythene bottles of 300 mL capacity. 100 mL of the solution containing 10 mg/L of Ni (II) ions under investigation adjusted to desired pH were taken in bottles and equilibrated for specific periods of time in a rotary mechanical shaker. At the end of equilibration period, the solutions were filtered using a G-3 crucible and the concentrations of Ni (II) ions were established by atomic absorption spectrophotometer (Elico, Model – SL 163). Adsorption isotherm and kinetic studies were carried out with different initial concentrations of Ni (II) by maintaining the modified carbon dosage at constant level. Ni (II) removal (%) was calculated using the following equation

$$\text{Removal (\%)} = \frac{C_i - C_f}{C_i} \times 100$$

Where  $C_i$  and  $C_f$  are the initial and final Ni (II) concentrations, respectively.

### Column studies

The investigation of column studies was done with sufficient weight of BCORC packed in a cylindrical tube of 2.5 cm diameter fitted with a Teflon stopper through the column. Optimum weight of carbon under proper flow rate and bed height conditions was used for column studies. An optimum flow rate of 10mL /min with the optimum bed height of

13.2cm was employed to assess the potential of BCORC carbon. Similarly other column of same diameter containing commercially available Activated Carbon (CAC) was packed under an optimum bed height of 6.8 cm and a flow rate of 6mL/min was maintained. The initial concentrations of Ni (II) employed were prepared in distilled water of 200mg/L for BCORC and CAC. Percolation of Ni (II) solution was stopped as soon as Ni (II) concentration in the effluent exceeded the arbitrary value of 1 mg/100mL (Breakpoint). 100 mL of lot volumes for Ni (II) content were analyzed by using atomic absorption spectrophotometer. Regeneration and recycling of BCORC carbon was done by treating with 0.5 N hydrochloric acid and thoroughly washed and reloaded in the column after drying. Breakthrough studies pertaining to Ni (II) adsorption in the presence of common impurities such as bicarbonate, chloride, sulphate, calcium and magnesium were done under optimum bed height and flow rate condition for both the carbons.

### Regeneration studies

In order to find out the percentage of Ni (II) desorbed, a series of experiments was conducted with both BCORC and CAC, by equilibrating with 100mL of 10mg/L of Ni (II) solution containing 100mg of BCORC and 200 mg of CAC carbon at an optimum pH of 6 and 8 respectively and these solutions were equilibrated for 24 h. At the end of equilibration period, the solution was centrifuged and analyzed for Ni (II) content. The carbons were then separated carefully, washed several times with distilled water to remove any unadsorbed Ni (II). To each

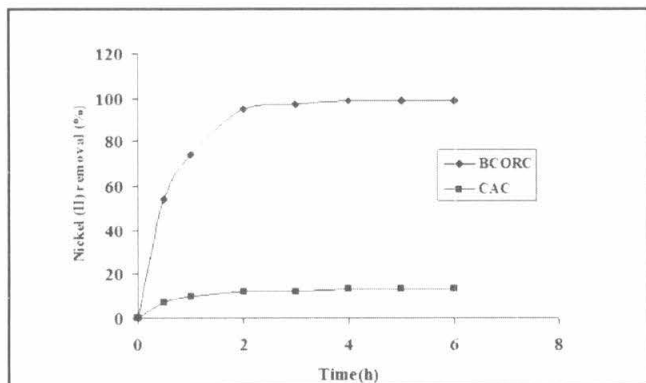
100 mL of hydrochloric acid over the range 0.1N -1.0 N was added and shaken for a period of 4h, in a mechanical shaker.

**Results and discussion**

Examination of carbon characteristics (Table1) shows that BCORC has considerable bulk density and high ion-exchange capacity. The moisture content of BCORC suggests that the acid treatment has made the carbon porous in nature. Even though BCORC shows lower surface area when compared with CAC, it has shown considerable ion-exchange capacity, through which the Ni (II) removal may also be taking place.

*Effect of agitation time*

Fig.1 shows the effect of agitation time on the removal of Ni (II) by BCORC and CAC. The removal increases with time and attains equilibrium in 4 hours for both BCORC and CAC for an initial Nickel (II) concentration of 10 mg/L and for a carbon dosage 100mg/100mL. In the case of BCORC 99% Ni (II) removal was achieved and in the case of CAC 13% Ni (II) removal was achieved.

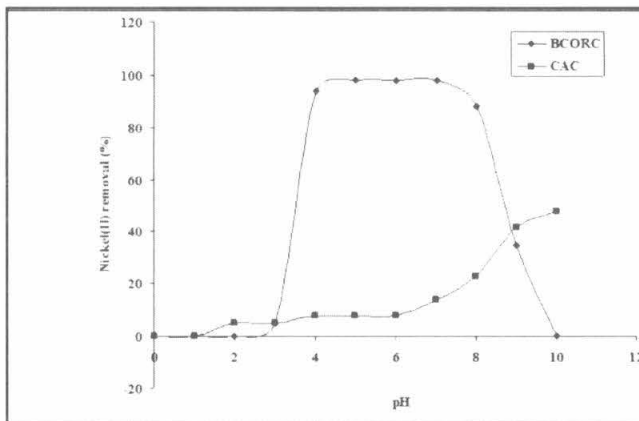


**Fig. 1 : Effect of contact time on the adsorption of Ni (II)**

*Effect of pH*

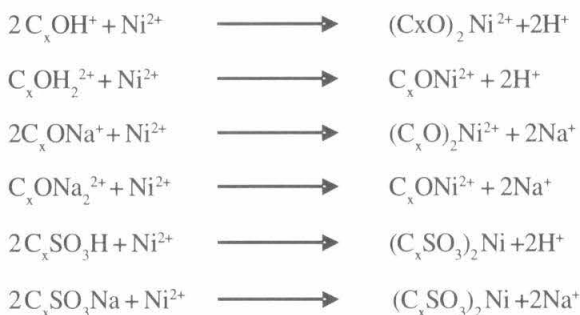
Fig.2 presents the effect of pH on the removal of Ni (II) by BCORC and CAC. It is evident that Ni (II) removal was 99% in the pH range 4.0 – 7.0 for BCORC and 42% in the pH range of 8-10.0 for CAC. BCORC is two times more effective in the removal of Ni (II) at an optimum pH of 6.0 than CAC at an optimum pH of 8.5. And also BCORC has a wide range of pH when compared with CAC.

The influence of pH on Ni (II) removal may be explained as follows. A pure carbon surface is considered to



**Fig. 2 : Effect of pH on the adsorption of Ni (II)**

be non-porous but in actual practice some carbon-oxygen complexes such as C<sub>x</sub>O, CO<sub>x</sub> and C<sub>x</sub>O<sub>2</sub> are usually present which render the surface slightly polar<sup>7</sup>. In the case of BCORC the following mechanism is suggested. Based upon the work of Frumkin<sup>4</sup>, the surface oxide groups available on the surface of a carbon can undergo hydrolytic reaction with the result proton exchangeable sites such as C<sub>x</sub>OH<sub>2</sub><sup>2+</sup>, C<sub>x</sub>OH<sup>+</sup> may be formed. Since CORC was prepared upon treatment with sulphuric acid followed by sodium bicarbonate soaking, groups such as C<sub>x</sub>ONa<sup>+</sup>, C<sub>x</sub>ONa<sub>2</sub><sup>2+</sup>, and C<sub>x</sub>SO<sub>3</sub>Na may be present. Hence it is expected that Na<sup>+</sup> in the group got exchanged with Ni (II) and soluble complexes of Ni (II) as follows.

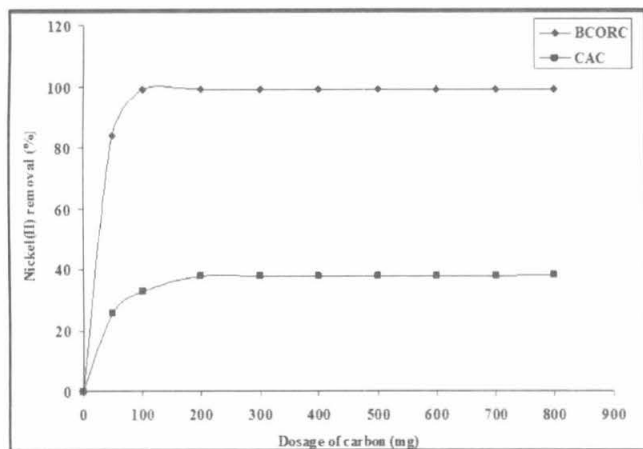


In order to confirm the ion-exchange mechanism the eluant collected after adsorption was subjected to analyse the amount of sodium by flame photometry method. The presence of considerable amount of Na present in the eluant confirms that release of Na<sup>+</sup> ions taking place during adsorption process.

*Effect of carbon dosage*

Fig.3 shows the removal of Ni (II) as a function of carbon dosage by both BCORC and CAC. It is evident that for

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**Fig. 3 :** Effect of carbon dosage on the removal of Ni (II)

99% removal of Ni (II) of 10mg/L of Ni (II) in 100mL, a minimum carbon dosage of 100 mg is required in case of BCORC and 37.8% of Ni (II) removal with 200 mg is required in the case of CAC. The data clearly indicated that BCORC is nearly 2 times more effective than CAC. This may be due to the moderate ion-exchange capacity of BCORC as compared to CAC.

### Adsorption isotherms

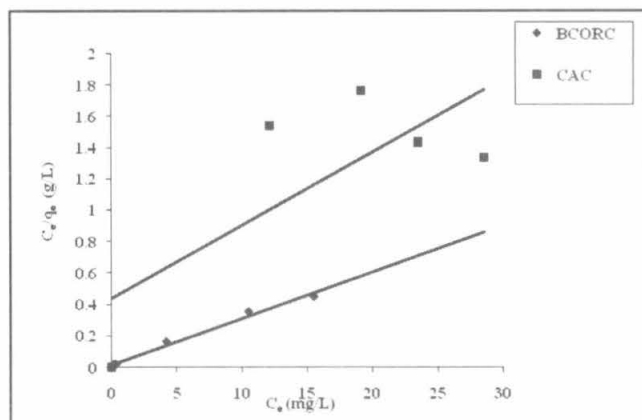
The Langmuir equation was applied for adsorption equilibrium for both BCORC and CAC.

$$C_e/q_e = 1/Q_0b + C_e/Q_0$$

Where  $C_e$  is the equilibrium concentration mg/L.  $q_e$  is the amount adsorbed at equilibrium (mg/g) and  $Q_0$  and  $b$  are Langmuir constant related to adsorption capacity and energy of adsorption respectively. The Linear plots  $C_e/q_e$  Vs  $C_e$  show that the adsorption obeys the Langmuir model for both BCORC and CAC and it is shown in Fig.4  $Q_0$  and  $b$  were determined from the Langmuir plots and found to be 25.00 mg/g and 2.0 mg/L respectively for BCORC and 16.666 mg/g and 0.0666 mg/L respectively for CAC. The ratio of  $Q_0$  values of BCORC and CAC works out to be 1.5. The essential characteristics of Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor (or) equilibrium parameter  $R_L$  which is shown as  $R_L = 1/(1+b C_0)$  where  $b$  is Langmuir constant and

**Table 3 :** K and n values of Freundlich adsorption isotherm

Carbon	Distilled water		Tap water	
	K (mg/g <sup>-1</sup> )	n (L mg <sup>-1</sup> )	K (mg/g <sup>-1</sup> )	n (L mg <sup>-1</sup> )
BCORC	15.0	3.50	4.0	1.66
CAC	0.16	0.7	0.1	0.60



**Fig. 4 :** Application of Langmuir model to the experimental data

$C_0$  be the initial concentration of Ni (II),  $R_L$  value shown in Table 2, indicates favorable absorption of Ni (II) on both BCORC and CAC at the concentration studies at room temperature  $30 \pm 1^\circ\text{C}$ . The Freundlich isotherm is represented by the equation. <sup>9</sup>

$$\log x/m = \log K + 1/n (\log C_e)$$

**Table 2 :**  $R_L$  values of BCORC and CAC for Ni (II) adsorption

Sl. No.	Concentration in mg/L	$R_L$ values	
		BCORC	CAC
1	10	0.0333	0.0938
2	20	0.0166	0.0475
3	30	0.0111	0.0322
4	40	0.0083	0.0243
5	50	0.0066	0.0188

Where  $C_e$  is the equilibrium concentration (mg/L) and  $x/m$  is the amount adsorbed per unit weight of BCORC (or) CAC (mg/g). Plots of  $\log (x/m)$  Vs  $\log C_e$  are linear for both BCORC and CAC. Fig.5 shows the Freundlich adsorption isotherm for BCORC and CAC, in tap water and distilled water. The process followed was of Freundlich adsorption type. The 'K' and 'n' values for both the carbon were calculated from the intercepts and slopes, respectively and are shown in Table 3. The values of  $1 < n < 10$  show favorable absorption of Ni (II) on both BCORC and CAC. <sup>9</sup>

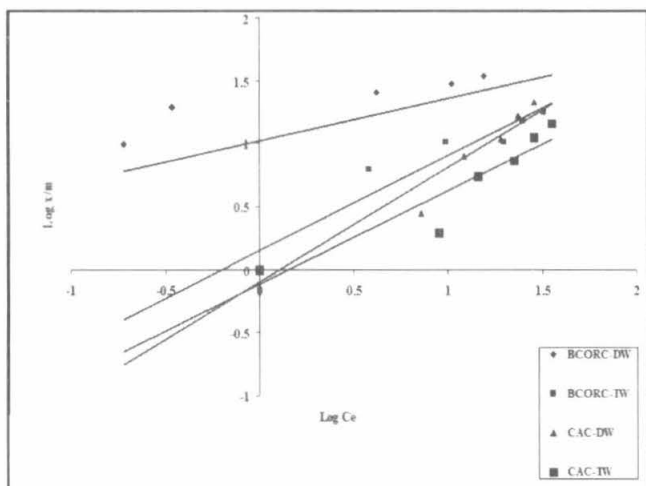
**Table 4 :** Rate constants for the removal of Nickel of BCORC and CAC

Name of carbon	Concentration of Ni (II) (mg/L)	K	$k_1$	$k_2$
		overall rate constant (h <sup>-1</sup> )	forward rate constant(h <sup>-1</sup> )	backward rate constant(h <sup>-1</sup> )
BCORC	10	1.444	1.438	0.0058
	7	2.000	1.828	0.1720
	5	2.222	2.2044	0.0180
	3	1.2220	1.812	0.0408
CAC	10	2.000	1.3514	0.5266
	7	1.7500	1.2999	0.4501
	5	0.5710	0.4111	0.1600
	3	0.7142	0.6327	0.0815

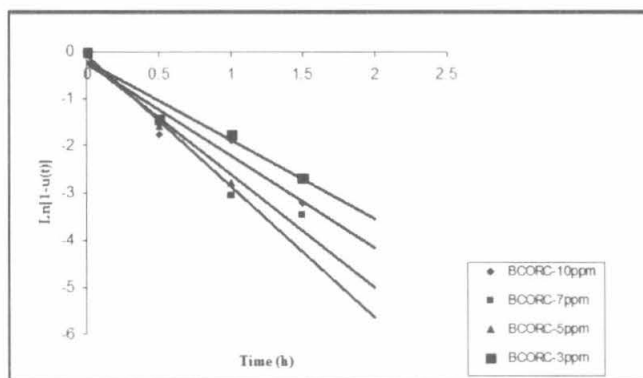
*Adsorption kinetics*

The kinetics of Ni (II) adsorption on both BCORC and CAC follows the first order rate expression. The kinetic fits for both BCORC and CAC are shown in Fig.6 and Fig.7. The detailed mathematical derivations for calculating the

reversible first order kinetics and the values of film and pore diffusion co-efficient are described elsewhere<sup>10</sup>. The kinetic data are furnished in Table 4. It is evident that the forward rate constant is much higher than the backward rate constant, suggesting that the rate of adsorption is clearly dominant for BCORC and CAC. According to Michelsen<sup>7</sup> for the adsorption of heavy metals on carbon surface, for film diffusion to be the rate determining process, the values of film diffusion co-



**Fig. 5 :** Application of Freundlich model to the experimental data

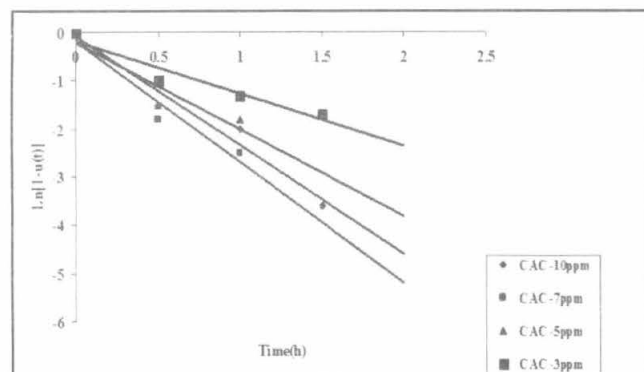


**Fig. 6 :** Kinetics for the adsorption of Nickel (II) in case of BCORC

**Table 5 :** Diffusion co-efficient for the removal of Ni (II) by BCORC and CAC

Concentration of Ni (II) mg/L	Diffusion co-efficient in cm <sup>2</sup> /s BCORC		Diffusion co-efficient in cm <sup>2</sup> /s CAC	
	film diffusion D <sub>f</sub>	pore diffusion D <sub>p</sub>	film diffusion D <sub>f</sub>	pore diffusion D <sub>p</sub>
10	1.5310 x 10 <sup>-10</sup>	1.435 x 10 <sup>-8</sup>	1.3782 x 10 <sup>-9</sup>	1.9879 x 10 <sup>-8</sup>
7	4.5437 x 10 <sup>-10</sup>	1.9878 x 10 <sup>-8</sup>	2.724 x 10 <sup>-9</sup>	1.7394 x 10 <sup>-8</sup>
5	4.7111 x 10 <sup>-10</sup>	2.2085 x 10 <sup>-8</sup>	4.2376 x 10 <sup>-10</sup>	5.6754 x 10 <sup>-9</sup>
3	3.1309 x 10 <sup>-9</sup>	1.2146 x 10 <sup>-8</sup>	2.1454 x 10 <sup>-10</sup>	7.0987 x 10 <sup>-9</sup>

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**Fig.7 : Kinetics for the adsorption of Nickel (II) in case of CAC**

efficient ( $D_f$ ) should be between  $10^{-6} - 10^{-8} \text{ cm}^2/\text{sec}$ . If pore diffusion ( $D_p$ ) were to be rate determining, its value should be in the range of  $10^{-11} - 10^{-13} \text{ cm}^2/\text{sec}$ . Using kinetic data, the values for film and pore diffusion constants were calculated and shown in **Table 5**. It is evident that the removals of Ni (II) follow film diffusion process as outlined by Michelsen<sup>7</sup>. Since the co-efficient values are very close to the range  $10^{-8} \text{ cm}^2/\text{sec}$ . It could be seen from **Table 5** that the Ni (II) adsorption was following film diffusion process on both carbons since

these values are much closer to film diffusion process. Based on the regeneration studies, it could be seen from **Table 6** about 87.00% and 25.8% of Ni (II) could be recovered from BCORC and CAC, respectively with a minimum concentration of 0.5 M Hydrochloric acid. Therefore it may be concluded that about 3 times of valuable Ni (II) could be recovered with BCORC. Based upon column studies **Table 7** indicates the breakthrough capacities of carbon under optimum flow rate and bed height conditions of BCORC and CAC in distilled water from which it could be seen that BCORC is about 14 times superior to CAC.

**Table 8** indicates the effects of common anions and cations, may be in water, on the removal of Ni (II) by BCORC and CAC. It could be seen that decrease in the capacities was noted in both BCORC and CAC. Bicarbonate and other anions and common cations such as calcium and magnesium ions affect the breakthrough capacity of CAC more than BCORC. The decrease in the removal of Ni (II) by both the carbons may be due to the formation of chloro and sulphate complexes of nickel and competition for the ion exchange sites by calcium and magnesium in the case of BCORC.

**Table 6 :** Desorption of Ni (II) with hydrochloric acid

Concentration of Hydrochloric Acid (M)	Recovery of Ni (II) from BCORC (%)	Recovery of Ni (II) from CAC (%)
0.05	58.7	16.31
0.1	77.0	16.51
0.15	83.0	18.02
0.2	85.3	22.13
0.25	86	24.56
0.3	86	24.56
0.35	86.36	25.32
0.4	86.3	25.8
0.45	87.0	25.8
0.5	87.0	25.8
1.0	87.0	25.4
2.0	87.0	25.4

**Table 7 :** Breakthrough capacity of carbons

Optimum flow rate (BCORC) – 10mL/min Optimum weight of carbon (BCORC)-20g		Optimum flow rate (CAC) -6mL/min Optimum weight of carbon (CAC) -15g	
Condition	Breakthrough capacity in mg		
Room temperature	BCORC 280.00	CAC 19.20	

**Table 8 :** Effect of common anions and cations on the removal of Ni (II) under optimum flow rate and bed height condition

Carbon	None mg/L	HCO <sub>3</sub> <sup>2-</sup> ions (1000mg/L)	Cl <sup>-</sup> ions (1000mg/L)	SO <sub>4</sub> <sup>2-</sup> ions (1000mg/L)	Ca <sup>2+</sup> ions (1000mg/L)	Mg <sup>2+</sup> ions (1000mg/L)
BCORC	280	220.0	240.0	199.94	59.95	60.0
CAC	19.20	19.00	17.22	18.85	17.87	16.08

**Table 9 :** Regeneration studies

Cycle	BCORC	CAC
I	299.92	NIL
II	319.97	Nil
III	280.00	Nil
IV	299.95	Nil
V	319.92	Nil

BCORC-10mL/min/20g at pH 6.0  
CAC- 6mL/min/15g at pH 8.0

**Table 10 :** Ni (II) wastewater characteristics

Parameters	Amount in mg/L (before treatment)	Amount in mg/L (after treatment)
Nickel(II)	216.3	33.95
Calcium	20.3	2.9
Magnesium	16.80	1.2
Chloride	78.9	18.4
Sulphate	41.13	13.4
pH 6.5	6.0	6.0

BCORC showed constant breakthrough capacity values in distilled water under different regeneration cycles indicating that carbon is effective in the removal of Ni (II) over the number of cycles and it was not undergoing any significant degradation in particle size because of its hardness. However, CAC capacity was very much affected by the different cycles and showed nil value for I to V cycle of operation shown in **Table 9**. **Table 10** indicates experiments with synthetic wastewater similar to that of Nickel electroplating industry wastewater used. BCORC showed the capacity value as 172.65mg/g and CAC showed nil capacity value.

**Conclusion**

Activated carbon prepared from coconut oilcake waste, generated from oil industries after extracting oil, is capable of removing Ni (II) effectively from aqueous solution. The bicarbonate treated material and commercial activated

carbon, conforms to Langmuir and Freundlich equation based on the formation of monolayer. The adsorption capacity of BCORC was found to be more and quantitative than commercial activated carbon based upon its wider pH range for adsorption of Ni (II) and also on the carbon dosage. The adsorption of Ni (II), on both the carbons follows first order reversible kinetics with film diffusion and ion exchange process being the essential rate controlling step. The kinetic data would be useful for fabrication and designing of wastewater treatment plants. Based upon desorption studies of Ni (II), it could be concluded that BCORC was found to be more effective than CAC with respect to adsorption and recovery of the metal. Column studies also demonstrated that BCORC showed better removal of Ni (II) in the presence of common impurities in water and wastewater when compared with CAC.

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**Symbols and abbreviations**

- ASTM - American Society of Testing Materials
- BCORC - Bicarbonate treated coconut oilcake residue carbon
- b - Langmuir constant (surface energy adsorption) (L mg<sup>-1</sup>)
- CAC - Commercial activated carbon
- C<sub>e</sub> - Metal ion concentration at equilibrium time (mg L<sup>-1</sup>)
- C<sub>i</sub> - Initial concentration of metal ion (mg L<sup>-1</sup>)
- C<sub>f</sub> - final concentration of metal ion (mg L<sup>-1</sup>)
- cm - centimetre
- cm<sup>2</sup>/sec - centimetre square per second
- D<sub>f</sub> - film diffusion co-efficient (cm<sup>2</sup>/s)
- D<sub>p</sub> - pore diffusion co-efficient (cm<sup>2</sup>/s)

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DW	- Distilled water
Fig.	- Figure
h	- hour
K	- Overall rate constant ( $h^{-1}$ )
$k_1$	- forward rate constant ( $h^{-1}$ )
$k_2$	- backward rate constant ( $h^{-1}$ )
$K_F$	- Freundlich constants related to adsorption capacity (mg/g)
L	- Litre
mg	- milligram
mg/g	- milligram per gram
mg/L	- milligram per Litre
m.equiv./g	- milli equivalents per gram
mL/min	- milliliter per minute
M	- Molar
Ni (II)	- Nickel (II)
n	- Freundlich constant (energy of adsorption) ( $L\ mg^{-1}$ )
ppm	- parts per million
$q_e$	- amount adsorbed at equilibrium time (mg per g)
q	- Amount of metal adsorbed at time, t (mg per g)
$Q_0$	- Langmuir constant (adsorption capacity) (mg per g)
$R_L$	- Equilibrium parameter or dimensionless constant
Sq.m/g	- square metre per gram
TW	- Tap water
(%)	- percent

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