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Activated carbon prepared from crushed pine needles used for the removal of Ni and Cd

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ABSTRACT

A new activated carbon (AC), prepared by carbonization and chemical activation of crushed pine needles, was characterized and examined for its potential in removing two heavy metals, namely Nickel (Ni) and Cadmium (Cd). Fixed-bed column testing procedure was adopted in the study. Depending on the individual metal tested, different initial solution pH values and different initial metal concentrations were used to evaluate their impacts on the AC adsorption process. Breakthrough curves were used to assess the adsorption capacities. Batch reaction tests were also conducted and the resulting data were fitted to adsorption isotherms. Furthermore tests were conducted where the two metals were combined in the test samples so as to determine their synergistic effect. The results indicated that the highest metal removals and adsorption capacities were attained at the highest pH values and concentrations tested for each of the two metals. Also, the results recorded for the batch experiments exhibited higher capacities than those recorded for the column tests and the resulting data fitted best the Freundlich isotherm. The results of the binary tests showed that the AC adsorption capacity of the individual metals was not affected.

Keywords: Activated carbon; Pine needles; Heavy metals removal; Wastewater; Adsorption

1. Introduction

The last decades have witnessed a large increase in the number of industries in the developing countries. Despite the concomitant benefits brought about by this rapid industrialization, a major negative impact associated with industrialization is the increase in environmental pollution. Industrial effluents and wastes usually contain high amounts of toxic heavy metal, such as, copper, cadmium, nickel, mercury, and lead amongst others [1,2]. It is anticipated that the demand for industrial products in the developing world will increase in future [3], which will result in more pollutants being emitted.

Adsorption technologies are known to be cost-effective and efficient methods for treating gaseous and liquid wastes. Due to their high surface area and versatile chemical properties, adsorbents have the

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ability to treat several types of industrial effluents and remove a wide range of organic and inorganic pollutants [4]. Activated carbon (AC) is a well-known adsorbent that has been used extensively in the treatment and removal of various heavy metals from industrial wastewater effluents. Hasar [5] reported that an AC prepared from almond husks, when tested on water spiked with Ni, could achieve a 97.8% nickel ion removal efficiency using 5 g/L of AC from an initial nickel ion solution of 25 mg/L [5]. Issabayeva et al. [6] showed that adsorption of Pb, from a solution with concentrations varying from 10 to 700 mg/L, on palm shell AC was higher at pH 5.0 than at pH 3.0. Corapcioglu and Huang [7] concluded from their study that with proper selection of the type of AC and operational conditions (pH, surface loading, and temperature) it is possible to have AC, which is as effective as other adsorbents such as iron and aluminum hydroxides, in removing the tested heavy metals (Cu(II), Pb(II), Ni(II), and Zn(II)). However the relatively high cost of ACs poses a disadvantage when it is to be used on a large scale. To alleviate this problem the search for cheaper precursor sources and production technologies as to arrive at reduced AC production costs is a necessity.

The objective of the study is to produce and characterize a newly introduced AC from crushed pine needles and evaluate its potential in adsorbing toxic heavy metals that include Ni and Cd. Using shed pine needles as the precursor provides a material which is widely available on a global level, is relatively easy to collect, handle, and convert into AC. All of these factors lead to a precursor that is cheaply available and sustainable. The study will report the results of tests conducted to determine the efficiency of the produced AC in removing the two metals, which are commonly discharged in water bodies from industrial sources. Selection of the metals is related to their wide use and consequently to their discharge through run-off from agricultural land and from various industries such as battery production, metal smelting, and fertilizer production (specific to Cd) [8], while the primary source for Ni discharge is from leaching in pipes and fittings and secondary sources such as wastewater industries involved in electroplating, leather tanning, wood preservation, and steel manufacturing. Concentrations of 5–30 mg/L and 30–50 mg/L are reported to reflect the concentrations of Cd and Ni in industrial wastewater, respectively [9–12]. Because of their health hazards, stringent standards were set for cadmium in drinking water by EPA at 0.005 mg/L, while for Ni the guideline concentration in drinking water was set at 0.07 mg/L [8,13].

2. Materials and methods

2.1. Preparation of AC

The AC used in this study utilizes pine needles as a precursor. Chemical activation was performed by impregnating the washed, dried, and crushed pine needles in KOH, with occasional mixing, for a period of 24 h. The mixture was then dried at 120˚C for another 24 h. The carbonization step was performed by placing the dried needle mixture, after containment in covered porcelain crucibles, into a furnace under an inert atmosphere (continuous nitrogen flow into the furnace at a rate of 500–700 mL/min). Heat was added at a rate of 10˚C/min intervals until a temperature of 800˚C was reached. The temperature was then held constant for a period of one hour. The product was left to cool down for several hours in the oven while maintaining the inert nitrogen flow control. After carbonization, the AC was washed in boiling distilled water, filtered, and washed with 5 M HCl. This was followed by carefully re-washing with distilled water to remove all the free acid. The washed AC was dried at 105˚C, crushed again after drying, and then stored in plastic vials and placed in a desiccator ready for use. Table 1 presents the characteristics of the AC product as well as the adopted analytical methods.

2.2. Fixed-bed adsorption column

The experimental setup is shown in Fig. 1, where two plexiglas columns 15 cm long and 2.5 cm in diameter are packed with thoroughly mixed batches of AC to ensure a uniform bed composition.

Each column was loaded with exactly 2 g of the AC. Prior to loading, the AC was boiled in distilled water for 10 min in accordance with ATSM method D6586 [26], so as to ensure that all the air inside the AC pores is replaced by water. Peristaltic pumps (master flex pumps manufactured by Cole-Parmer, USA) were used to ensure constant flow of the solution to the top of the column, where the flow through was effected by gravity. Flow rates into the column were varied between experiments in accordance with the set experimental plan. The columns were packed at the bottom with glass wool to ensure that AC particles will not be transported out of the bottom of the column. After each test the columns were acid washed.

Stock solutions of metal were prepared in concentrations ranging between 200 and 300 mg/L, and later dilutions were made depending on the requirements of the experiments. Based on need, the pH of the diluted solutions was adjusted using 0.5 M HCl or 0.5 M NaOH.

Table 1
Parametric values and methods used in characterization of the pine-needle AC

Characteristic	Value	Method	Machine(s)used	Source
Apparent density	158 g/L	ASTMD-2854		[14]
Iodine number	962.9 mg/g	ASTMD-4607	HACH DR/2010 Spectrophotometer	[15]
Moisture content	0.57%	ASTMD-2867	RAYPA,Vacuum Oven Model EV-50	[16]
pH value	5.2	ASTMD-3838	Orion Research pH meter 811	[17]
Total ash content	20.88%	ASTMD-2866	NABERTHERM, Oven ModelL40	[18]
Volatile matter content	44.93%	ASTMD-5832	NABERTHERM, Oven ModelL40	[19]
Surface and porosity imagery		Scanning electron microscope	TESCANSEM	[20,21]
Surface area per volume	854 m^2/g	Nitrogen adsorption	Nova 2200e surface area and pore size analyzer	[22]
Particle density and porosity	1.4 g/mL	Water displacement		[23]
Methylene blue number	443 mg/g	MB adsorption	HACH DR/2010 Spectrophotometer	[24]
Surface functional groups		FTIR	NICOLET 4700 FT-IR	[20]
Surface charge	3.12	PZC determination	Orion Research pH meter 811	[25]

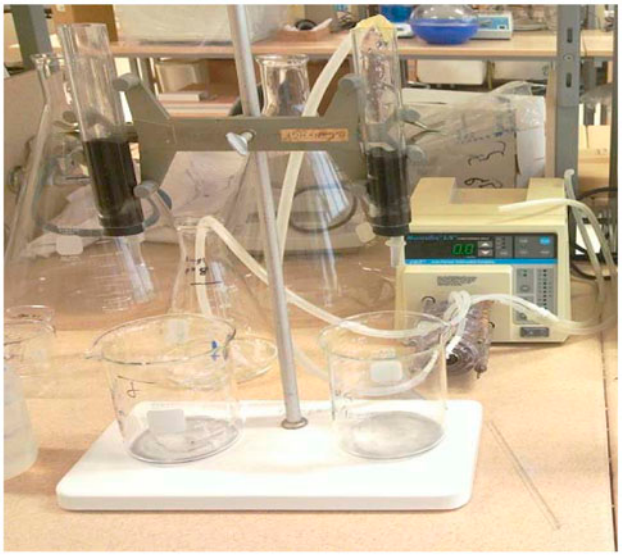


Fig. 1. View of the experimental setup for fixed-bed adsorption columns.

Influent metal concentration was constantly measured after fixing the pH. Periodic sampling of the effluent was done based on the metal adsorption rate. Sample collections as close as one minute apart were taken for some, while others were as far as 30 min apart. These samples were collected in acid washed plastic tubes so as to be directly used with the flame atomic absorption spectrophotometer.

Selecting the pH to be adopted in the study was based on the metal hydroxide solubility in solution. Both Cd and Ni become almost insoluble in water above pH 8, thus testing was restricted to pH values less than eight with the lower limit set at pH four or five based on practical applications. The array of tests conducted covered pH values 5, 6, 7, and 8 and concentrations of 1 and 5 mg/L for Cd and at pH values of 4, 5, 6, 7, and 8 and concentrations of 2 and 6 mg/L for Ni. Duplicates or triplicates of each test were conducted to ensure the reproducibility of the results.

Parametric analytical procedures for pH and metal concentration were based on Standard Methods [27] using methods 4500-H^+ for pH value and method 3111 for Cd and Ni, respectively. pH was measured using Orion research pH meter model 811 while metal concentrations were measured by using an AA spectrophotometer manufactured by Thermo Electron Corporation (USA).

2.3. Batch reaction adsorption tests

For the purpose of determining adsorption isotherms, batch reaction tests were carried out at constant pH, adsorbent concentration, and at room temperature (22 ± 2˚C). Adsorbent concentration was set for all experiments at 4 g/L. The selected pH was that at which optimal removal was detected from the column experiments. Metal concentrations were varied. For each metal solution a 100 mL aliquot was placed in 125 mL conical flasks and 0.4 g of pine-needle AC was added to each flask. These flasks were constantly stirred starting at 150 rpm for the first four hours, then at 100 rpm until equilibrium was reached. Batch reactions for Cd and Ni were conducted at pH of 8 (fixed after addition of AC). The concentrations for Cd varied from 5 to 200 mg/L and for Ni from 5 to 350 mg/L. At the end of the first run of the batch experiments, it was noted that the concentration of Ni remaining in the beakers, that had low initial concentration, approached zero. This meant that the data cannot be fitted to the adsorption isotherms. It was decided to increase the upper limit of the initial Ni concentration to reach up to 350 mg/L.

3. Results and discussion

3.1. Cadmium removal

The effect of the initial pH of the solution was studied at the range of 5–8, and at two different metal concentrations of 1 and 5 mg/L. Fig. 2(a) and (b) present the measured results at the concentration of 1 mg/L and 5 mg/L, respectively, while Table 2 indicates the adsorption capacities of the pine-needle AC for cadmium removal.

The figures indicate that at both concentrations the increase in pH leads to an increase in the removal capacity of the pine-needle AC reaching to a maximum removal at pH 8 under the study conditions.

The effect of change in initial solution concentration on removal capacity of Cd was studied at a concentration range of 1–40 mg/L and a pH of 8 as determined by the previous experiment. The results are shown in Fig. 3 and Table 3.

From the above results it may be surmised that the capacity removal rate of Cd is higher at lower initial concentrations, showing a maximum at a concentration of 5 mg/L then decreasing with the increase in concentration up to 40 mg/L.

As noted earlier, batch reaction experiments were conducted at the stated values (see section 2.3) and the data obtained was fitted to three isotherms (see Table 4).

Table 2
Adsorption capacity of the AC for Cd removal at different pH values

Capacity (mg/g)		
pH	Concentration	
	5 mg/L	1 mg/L
5	7.16	5.79
6	7.91	7.87
7	16.86	11.57
8	17.54	15.08

Where $q_{\max}$ and $q_{\exp}$ are in mg/g and are the monolayer capacity of the adsorbent and the amount of ions adsorbed at equilibrium, respectively, K_L is the adsorption equilibrium constant in L/mg, R^2 is the correlation factor, R_L is the separation factor, n is the heterogeneity factor, K_f is the Freundlich constant in L/g and a and b are Temkin constants. The data indicates that the removal mechanism fits almost perfectly the Freundlich isotherm where the batch reaction for Cd removal indicates an experimental value for $q_{\exp}$ to be equal to 43.47 mg Cd/g AC.

Fig. 4(a) shows that the removal capability of the pine-needle AC vs. the initial concentration of Cd in solution, while Fig. 4(b) shows the change in Cd removal vs. time. It is noted from Fig. 4(a) that the removal ability of the AC for Cd increases, almost linearly, with increasing concentration to reach a maximum at the highest tested concentrations. The fact that no plateau was reached may imply that the real removal capacity of the pine-needle AC for Cd is even higher than reported. Fig. 4(b) indicates that the high removal of Cd occurs very rapidly, for all concentrations, within the first 30 min. At lower Cd concentration, 5 mg/L, the maximum removal is

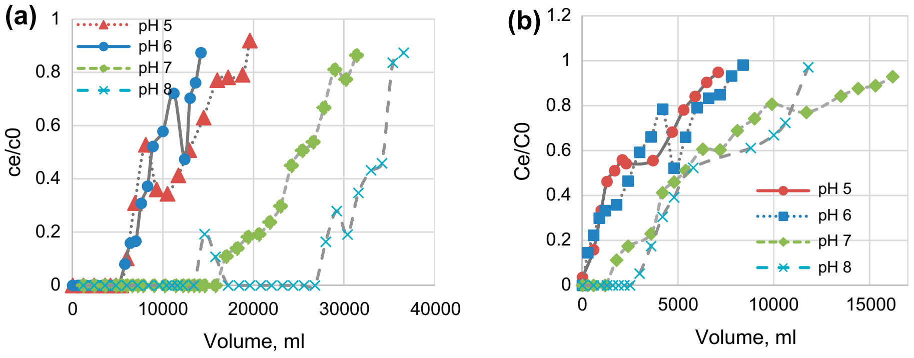


Fig. 2. Breakthrough curves for cadmium removal at varying pH and initial concentration of (a) 1 mg/L and (b) 5 mg/L.

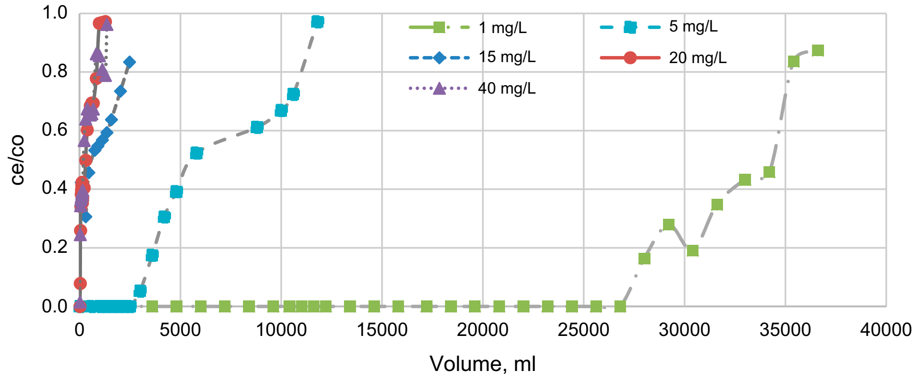


Fig. 3. Breakthrough curves for cadmium removal at varying initial concentration and pH 8.

Table 3
Adsorption capacity of the pine-needle AC for Cd at various concentrations

pH 8	
Initial concentration (mg/L)	Capacity (mg/g)
1	15.12
5	17.54
15	7.3
20	3.9
40	8.08

reached much later at equilibrium, when the solution was in contact with the AC for the longest period.

Cadmium removal by ACs has been investigated by several researchers at different conditions and with varying results. Some of these results are shown in Table 5 along with the results obtained in this study where it can be seen that the pine-needle AC possess excellent capabilities for Cd removal when compared to other types of AC.

3.2. Nickel removal

The effect of the initial pH of the solution was studied for the range of 4–8 and at two different Ni concentrations of 2 and 6 mg/L where the results are shown in Fig. 5. Table 6 indicates the Ni adsorption capacities of the pine-needle AC.

The data clearly indicate that, for the pH range investigated in this work, Ni is best removed by the pine-needle AC at a pH of 8. At an initial concentration of 6 mg/L, removal capacity of the AC improved by 67% when the pH increased from 4 to 5.

Table 4
Langmuir, Freundlich, and Temkin parameters for adsorption of Cd onto pine-needle AC

Langmuir parameters				Freundlich parameters			Temkin parameters		
$q_{\max}$	K_L	R^2	R_L	n	k_f	R^2	a	b	R^2
15.89	0.0504	0.5375	0.1–0.8	1.62	0.9029	0.9673	1.67	− 6.4248	0.8282

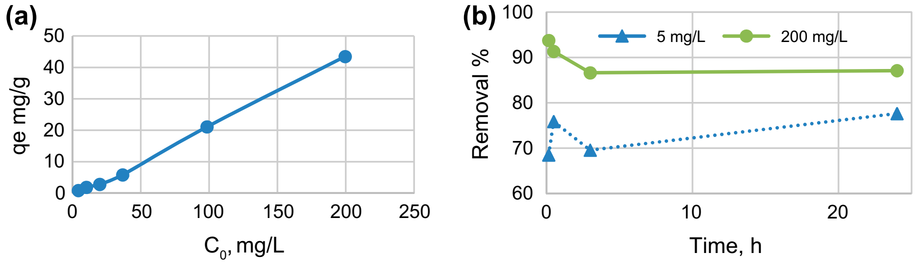


Fig. 4. Cadmium removal: (a) removal capacity for Cd vs. initial concentration and (b) Removal % of Cd vs. time.

Table 5
Comparison of Cd adsorption capacity of the pine-needle AC with other types of laboratory prepared ACs

AC type	Capacity (mg/g)	Experiment conditions	Source
Sugar-cane	$q_{exp} = 24.7$	AC dose of 2 g/L, pH 6, Initial[Cd] = 50 mg/L	[28]
Coconut	$q_{exp} = 11$	AC dose of 20 g/L, pH 6, Initial[Cd] = 500 mg/L	[29]
Bagasse	$q_{exp} = 38.0$	AC dose of 6 g/L, pH 4.5, Initial[Cd] = 200 mg/L	[30]
Peanut shell	$q_{exp} = 26.01$ $q_{max} = 48.7$	AC dose of 3 g/L, pH 4.8, Initial[Cd] = 550 mg/L − 5.5 g/L	[31]
Palm shell	$q_{max} = 13.05$	pH 3 and 5	[32]
Apricot stone	$q_{max} = 33.4$	pH 3 and 5	[32]
Bituminous coal	28.7	Column exp: Influent Cd 1 mg/L, 10 g AC, 3 cm/min	[33]
Pine needle	17.54	Column exp: pH 8, Influent Cd 5 mg/L, 2 g AC—20 mL/min	This study
Pine needle	$q_{exp} = 43.47$	AC dose of 4 g/L, pH 8—Initial[Cd] = 5–200 mg/L	This study

It then remained constant over the range of pH 5–7, and then it almost doubled when the pH increased from 7 to 8. For a concentration of 2 mg/L, the increase in pH from 4 to 5 resulted in doubling the removal capacity. A very large increase in AC capacity occurred when the pH increased from 5 to 6. It is worth noting that a dip in removal capacity happens when the pH changes from 6 to 7, and this is consistent for both concentrations (12.5% dip at 6 mg/L, and 16.4% dip 2 mg/L).

The above results indicate that for the conditions considered in this work, the best removal capacity of Ni is obtained at pH 8. Therefore, this pH value was used when testing for the effect of change in initial Ni concentration in the solution. Fig. 6 and Table 7 show the results of these experiments.

No specific pattern which relates the increase in initial Ni concentration to the AC's removal capacity can be concluded from the figure above. However, a large drop in adsorption capacity, by 59%, is noticeable when the initial Ni concentration increases from 20 to 40 mg/L. The highest capacity of the pine-needle AC is equally obtained at an initial concentration of 6 and 20 mg/L.

As noted earlier, batch reaction experiments were conducted at the stated values (see section 2.3) and the data obtained was fitted to three isotherms as shown in Table 8.

The experimental data showed that the maximum adsorption of Ni by pine-needle AC occurred at the highest tested concentration and reached 87.03 mg/g.

The rate of removal of Ni from solution and the effect of initial Ni concentration on the removal efficiency as determined from batch reaction data are shown in Fig. 7(a) and (b), respectively.

Fig. 7(a) indicates that the highest removal % was obtained during the first few minutes of the reaction. The equilibrium concentration was higher (by 5–10%) than the concentrations reached during the first few minutes. Fig. 7(b) indicates that the pine-needle was able to reach higher removals when the initial Ni concentrations were higher. At very low initial concentration (5–10 mg/L) the removal was below 80%; this increased gradually with the increase in concentration,

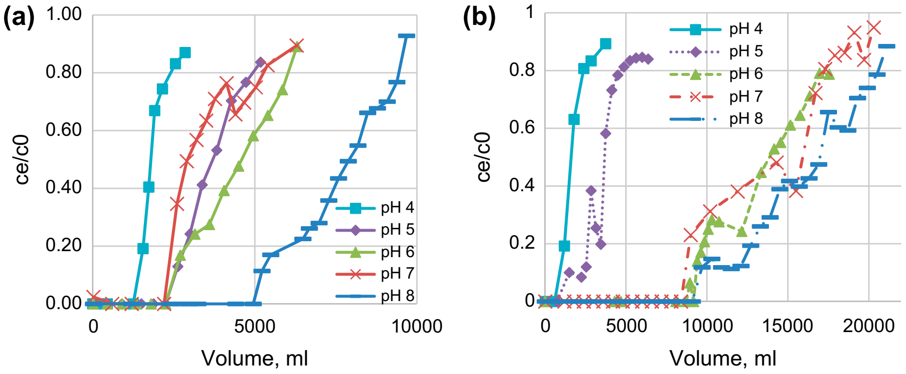


Fig. 5. Breakthrough curves for Cd removal at varying pH and initial concentrations of (a) 2 mg/L and (b) 6 mg/L.

Table 6
Adsorption capacity of the pine needle AC for Ni at various concentrations

Capacity (mg/g)		
pH	Concentration (mg/L)	
	6	2
4	6.57	1.7
5	10.98	3.76
6	11.82	13.73
7	10.34	11.48
8	20.85	16.6

to reach around 100% removal at concentrations of 130 and 350 mg/L. It is worth noting that for all of the experiments conducted, removal at the initial concentration of 200 mg/L was consistently lower than the removal at 130 and 350 mg/L, this was the only point that did not fit into the obtained trend.

Table 9 shows a comparison between the performance of pine-needle AC and a sample of other lab prepared AC in removing Ni at each AC's optimal conditions. Pine-needle AC possesses a remarkable ability to remove Ni compared to other laboratory produced ACs. Although some of the other ACs exhibit a better removal capacity, it should emphasize these ACs were tested under conditions that may increase the apparent ability of the AC to adsorb Ni, such as higher initial capacity in batch reactions, much larger column size and much slower flow rate through the column.

3.3. Binary system

In water or wastewater streams, several pollutants will be present in the solution. The presence of several contaminants will have a major effect on the ability of the AC to remove the metals, mainly due to the increased load, the interactions between the contaminants and the aqueous solution and because of the increased ionization of the solution.

Table 7
Adsorption capacity of the pine-needle AC for Cd at various concentrations

pH 8	
Initial concentration (mg/L)	Capacity (mg/g)
2	16.6
6	20.85
10	13.88
20	20.41
40	8.4

Tests were performed where Ni and Cd were present at the same time at an initial concentration of 5 mg/L each. The parameters and results of the experiments are shown in Fig. 8.

The breakthrough points of both metals are very close, this indicates that in a binary system, the pine-needle AC does not have a preference towards any of the metals, and it adsorbs both equally. However, after the start of the breakthrough, effluent concentration of Cd reached exhaustion concentration much faster than that of Ni. Furthermore, the capacity of the AC for Ni and Cd does not appear to be affected by the presence of a second metal in solution. Previous tests at similar conditions, although not the exact conditions, revealed that the AC's capacity for Ni is at 20.85 mg/g, which has slightly increased to 22.94 mg/g in the multi-component system. Also, the previously tested Cd removal capacity of the AC is 17.54 mg/g, which showed a noticeable increase during testing in the multi-component system. This increase in the capacity of the AC may be due to increased repulsion

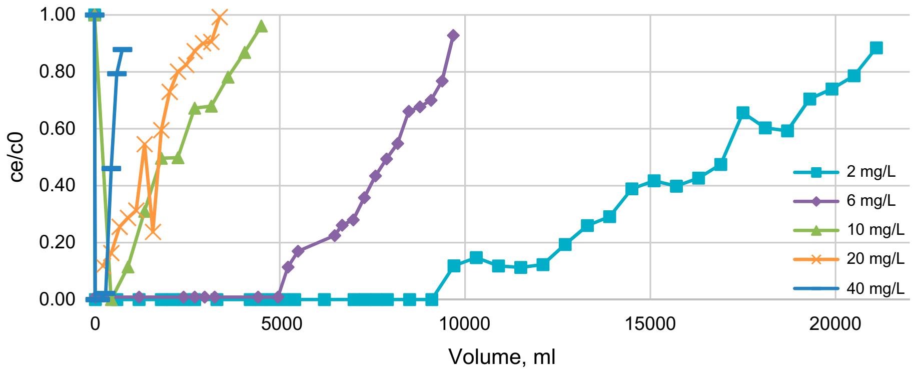


Fig. 6. Breakthrough curves for cadmium removal at varying concentration and initial pH of 8.

Table 8
Langmuir, Freundlich, and Temkin constants and correlation coefficients for adsorption of Ni

Langmuir parameters				Freundlich parameters			Temkin parameters		
q_{max}	K_L	R^2	R_L	n	k_f	R^2	a	b	R^2
77.52	0.089	0.2629	0.03–0.78	0.7	2.141	0.7157	2.627	13.567	0.2147

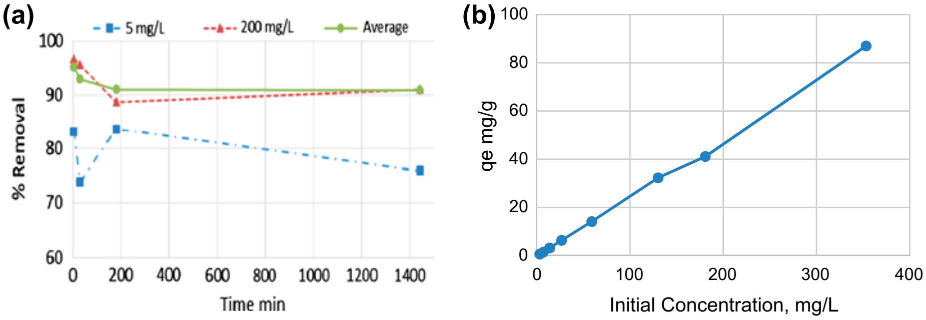
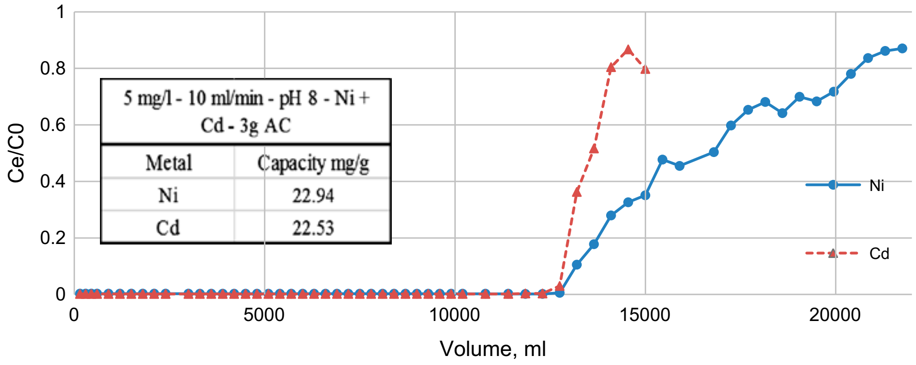


Fig. 7. Effect of different Ni concentrations on AC adsorption (a) (%) removal vs. time and (b) adsorption capacity.

Table 9
Comparison of the Ni adsorption capacity of the pine-needle AC with other lab prepared ACs

AC type	Capacity (mg/g)	Experiment conditions	Source
Apricot	101.01	AC dose of 70 g/L, pH 5, Initial[Ni] = 10	[34]
Almond husk	4.89	AC dose of 5 g/L, pH 5, Initial[Ni] = 25 mg/L	[3]
Tea waste	11.17	Column exp: Influent Ni 200 mg/L, 30 cm AC, 10 mL/min	[35]
Coirpith	$q_{max} = 62.5$	AC dose of 0.2 g/L, pH 5, Initial[Ni] = 10–40 mg/L	[2]
Coconut	14.64	AC dose of 20 g/L, pH 6, Initial[Ni] = 500 mg/L	[29]
Sugarcane	$q_{max} = 140.85$	AC dose of 2 g/L—pH 6.5—Initial[Ni] = 50–1,000 mg/L	[36]
Sugarcane	137.24	Column exp: Influent Ni 50 mg/L, 50 cm AC, 5.4 mL/min	[36]
Pine needle	20.85	Column exp: Influent Ni 6 mg/L, 2 g/10 cm AC, 20 mL/min	Current study
Pine needle	$q_{exp} = 87.03$	AC dose of 4 g/L, pH 8, Initial[Ni] = 350 mg/L	Current study



5 mg/l - 10 ml/min - pH 8 - Ni + Cd - 3g AC

Metal	Capacity mg/g
Ni	22.94
Cd	22.53

Fig. 8. Breakthrough curves for Ni and Cd in a binary system.

between the ions in solution which favors their movement towards the surface of the AC. It may also be due to the fact that adsorbing one metal onto the surface of the AC will improve its capacity towards removing other metals [37,38].

4. Conclusions

The ability of a newly prepared pine-needle AC to adsorb two heavy metals was tested under various operating conditions which were designed to replicate, industrial conditions. pH of solution proved to have a major impact on the adsorption capacity of the AC. In general, it was observed that an increase in solution pH led to an improvement in the removal capacity of the AC. The initial concentration of heavy metals had a clear effect on the adsorption capacity of the AC for metals. In batch experiments higher concentrations always resulted in higher removal and higher adsorption capacity. In column testing, high concentrations (40–50 mg/L) overwhelmed the capacity of the AC for removal and resulted in lower adsorption capacities. In fixed columns, Cd was better removed at the lower concentrations (5 mg/L), while Ni removal was almost stable in the lower and middle ranges, and dropped significantly when the initial concentration was at the maximum value of 40 mg/L. For Cd, the highest capacity in column testing reached 17.54 mg/g, and a much higher removal capacity was obtained in batch experiments, reaching 43.47 mg/g. The highest Ni capacity in column testing was 20.85 mg/g, while a much higher removal capacity of 87.03 mg/g was reached in batch experiments. In batch experiments, the Cd removal mechanism was best described by the Freundlich isotherm, while for Ni the results were not conclusive. For a binary system where both metal are present at the same time, the removal capacity of the AC was not affected, and its capacity reached almost 23 mg/g for each of the metals, which is a slight improvement compared to the unary systems.

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