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RESEARCH ARTICLE

Utilization of Charred Water Hyacinth (Jalkumvi) as Biosorbent for Removal of Ca(II) Ion from Aqueous Solution

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Abstract

A promising adsorbent, charred water hyacinth (CWH) for the removal of Ca(II) from the aqueous solution was explored by heat treatment of water hyacinth followed by chemical activation with acidified zinc chloride ($ZnCl_2$). The adsorbent was characterized using scanning electron microscopy (SEM) and electron dispersive X-ray (EDX) spectroscopy. Batch adsorption techniques were conducted for Ca(II) adsorption to assess the adsorption isotherm, effect of pH, contact time, initial Ca(II) concentration, adsorbent doses, and adsorption kinetics. The SEM micrograph illustrates the rough and irregular surface morphology and EDX spectra confirm the successful adsorption of Ca(II) on the adsorbent surface. The equilibrium adsorption data better fitted to the Freundlich isotherm model having a maximum adsorption capacity (q_{max}) of 319.75 mg/g. The highest percentage of adsorption was found at pH 1.5. The adsorption of Ca(II) by CWH decreased at the higher metal concentration and lower adsorbent doses. The adsorption of Ca(II) ions onto CWH followed the pseudo-second-order kinetics model. Overall, these results suggested that the as-prepared CWH can be used as an eco-friendly, economical and efficient alternative for the removal of Ca(II) from the aqueous solution.

Keywords: Charred water hyacinth, Freundlich adsorption isotherm, pseudo-second-order kinetics, SEM, EDX

Introduction

Calcium, considered as an important determinant of water hardness and a pH stabilizer, is abundant due to its natural occurrence in Earth crust in the form of limestone, marble, calcite, dolomite, gypsum, fluorite, and apatite and occurs as $Ca(OH)^+$ (aq), $Ca(OH)_2$ (aq) or as $CaSO_4$ in seawater [1]. Calcium is also abundant in the human body present it amount of 1.2 kg may be found as calcium phosphate supporting substance causing bone and tooth growth along with vitamin D [2]. Recently, increasing concerns have been raised regarding the increased amount of calcium in drinking water which causes the increment in the hardness of water [3]. Some recent studies have revealed that the excessive intake of calcium might increase the risks of cardiovascular diseases [4], growth reduction and reproductive failure [5], the buildup of lime-scale that fouls plumbing and promotes galvanic corrosion [6], influence metabolism, and affects kidney performance [7].

Calcium can be removed by ion-exchange [8], nano-filtration [9], ultrafiltration and reverse osmosis [10] processes which are economically expensive and are incomplete methods. Regarding these shortcomings, adsorption is considered to be an efficient and environmentally friendly method. Adsorption is the surface phenomena that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic film (adsorbate) [11,12].

Activated carbon is the most popular adsorbents for the removal of metal ions due to its highly developed porosity, large internal surface area, and relatively high mechanical strength. Its adsorbing potential is affected by the surface charge, type of surface functional groups, specific surface area, and pore size distribution [13]. The powdered activated charcoal can be obtained efficiently from various agricultural waste materials like coconut husk, peat moss, wool, rice husk, exhausted coffee, etc. which is a potential adsorbent for adsorption separation of calcium [14]. The low-cost adsorbents with improved sorption capacities derived from agricultural wastes for the removal of metallic and non-metallic pollutants have been the center of study for researchers [15-17].

In this case, we study the adsorption capacity of

Ca(II) on water hyacinth, a free-floating perennial aquatic plant of family *Pontederiaceae,* commonly known as Jalkumvi in Nepali, extracted from Begnash Lake, Kaski. The common water hyacinth (*Eichhornia crassipes*) is a noxious weed that has attracted worldwide attention due to its fast spread and congested growth, which lead to serious problems in navigation, irrigation, and power generation [18,19]. In Begnash Lake, water hyacinth is extracted during the growing season. The extracted raw materials are thrown as waste at the corner of the lake.

The conventional techniques used for the removal of Ca(II) from water are expensive for developing countries like Nepal. There are voluminous research works available in the literature regarding the use of waste biomass as an adsorbent for the removal of heavy metals [19,20], chromium [12,14,21,22], arsenic [23-25], phosphate [15,26], and Ca(II) [3,6,7] from aqueous medium. To the best of our knowledge, water hyacinth is a novel material and not studied yet for the removal of Ca(II) from water. Because of such circumstances, CWH is supposed to be a novel material herein studied and its application for removal of Ca(II) was proposed.

The objective of the present study was to prepare charred water hyacinth (CWH) by heat treatment of water hyacinth at 400^{o}C followed by chemical activation with acidified 0.1 M $ZnCl_2$ and to investigate its adsorption behaviors of Ca(II) from aqueous solution under different conditions through batch experiments. The adsorbent was characterized by SEM and EDX. The adsorption isotherm, effect of pH, contact time, adsorbent doses, adsorption kinetics on Ca(II) adsorption were also studied and discussed.

Materials and Method

Chemicals and analysis

The adsorbent used for this study is the leaves of the water hyacinth. The water hyacinth was collected from the Begnas Lake of the Kaski district. Analytical grade chemicals were used without additional purification. All the solutions in the work were prepared by using deionized water. EDTA ($C_{10}H_{16}N_2O_8$, 99% assay), hydrated magnesium chloride ($MgCl_2.6H_2O$, 97% assay), calcium carbonate ($CaCO_{3,}$ 98.5% assay), ammonium chloride (NH_4Cl) were brought from local suppliers, manufactured from Thermo Fischer Scientific India Pvt. Ltd. (India). A standard stock solution of Ca(II) (1000 mg/L) was prepared by dissolving an exact amount of $CaCO_3$ in deionized water. Ca(II) concentration in the test solution before and after adsorption was analyzed spectrophotometrically using ELICO SL 177 spectrophotometer.

Preparation of adsorbent

Thus collected raw water hyacinth was washed with tap water followed by distilled water and dried in sunlight for about 2-3 days. The carbonization was then conducted in a muffle furnace at 400^{0}C for 3 h in air. After carbonization, the residual char was grounded using a laboratory jar mill to pass between 25-50 mesh sizes.

Charred water hyacinth (CWH) was chemically activated with the help of acidified 0.1 M $ZnCl_2$ at laboratory temperature. The grounded char was mixed with $ZnCl_2$ solution of 1:2 ratio in porcelain. The sample was then activated in a muffle furnace at 600^{o}C for 3 h for complete reaction. The mixture was washed with 1% HCl and soaked in a hot water for 5 min and then dried at 120°C for 6-8 h in an oven. Finally, heat-treatment followed by chemical activation of raw water hyacinth gave activated biochar of water hyacinth [18]. Due to this chemical activation, the surface area of the adsorbent is expected to be increased which increases the adsorption sites [27,28].

Determination of λ_{max} and calibration curve

A Ca(II) ion solution of 25 ppm concentration was prepared for the determination of λ_{max} and calibration curve. For this 0.0, 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, and 7.0 mL of working Ca(II) solution of 20 mg/mL were taken in a different 25 mL volumetric flask and was acidified with 1 mL of 5 N H_2SO_4 in each volumetric flask and shaken well. In each volumetric flask, 1.0 mL of 0.001 M EDTA solution was added and shaken well. The remaining portion of the volumetric flask was filled with the distilled water. The absorption spectra of pinkish-blue colored calcium with EDTA complex was recorded with the help of a spectrophotometer (ELICO SL 177) against reagent blank solution. Thus, obtained adsorption data was plotted to find the calibration curve and absorption spectra for the determination of Ca(II) ions after the formation of a complex with EDTA at λ_{max} 520 nm [12].

Batch adsorption studies

The working solutions of required concentrations of Ca(II) ion were prepared to dilute the stock solution. The pH of the solution was adjusted by using acidified 0.1 M $ZnCl_2$ and 2N NaOH solutions. 25 mL of the known initial concentration of Ca(II) was taken in a

125 mL reagent bottle and predetermined about 15 mg of charred water hyacinth was added. The mixture in the reagent bottle was shaken for 24 h in a mechanical shaker at room temperature. The equilibrium was established. Thus obtained equilibrated solution was filtered with the help of filter paper and filtrate was used to analyze the remaining concentration of the metal ions, concentration before and after adsorption was determined by spectrophotometer at λ_{max} of 520 nm [29].

Batch pH studies

Batch pH studies were performed by shaking 25 mL of 20 mg/L of Ca(II) individual solution with 25 mg of the adsorbent for 24 h, to ensure the attainment of equilibrium over a wide range of initial pH values from 1.0 to 6.0. The samples were taken in 125 mL reagent bottles with an air-tight stopper. The equilibrium pH of the reaction mixture was recorded and the filtrate was used for the analysis of the remaining Ca(II) ion concentration. The experiments were conducted three times and the mean value was taken for the analysis of data. The optimum pH values were found out for the sorption of Ca(II) ions onto these adsorbents, which provides fruitful information for further study.

Batch equilibrium time studies

The equilibrium time of the adsorbents for the sorption of Ca(II) ion was investigated at optimum pH values at room temperature. 25 mL of 20 mg/L concentration of Ca(II) solutions were taken in 125 mL reagent bottle with 25 mg adsorbents and was shaken in a mechanical shaker for 10 min, 20 min, 30 min, 40 min, 50 min, 60 min, 70 min, 80 min, 90 min, 100 min, 2 h, 4 h, 5 h, 6 h, and 24 h, respectively. The metal ion concentration was determined by using ELICO SL 177 spectrophotometer.

Batch kinetic studies

The adsorption kinetic experiments were carried out at optimum pH for Ca(II) ion adsorption by equilibrating 25 mg of adsorbents in 125 mL stopper bottles containing 25 mL of 20 mg/L Ca(II) solution in each. Separate sets of various samples were kept on a mechanical shaker and shaken vigorously. Samples were filtered through filter paper and concentration before and after the adsorption was determined by the spectrophotometric method by analyzing the filtrate. The obtained data was tested with the pseudo-second-order and second-order kinetic model [30].

Effect of concentration of Ca(II) ions

Adsorption isotherm studies were conducted by varying the initial concentration of Ca(II) ions from 10 to 500 mg/L. 25 mL of different concentrations of Ca(II) solution were shaken by the mechanical shaker for 24 h with 25 mg of adsorbent dose for both of the samples raw water hyacinth (RWH) and charred water hyacinth (CWH). The remaining Ca(II) ion concentration was determined after filtering the reaction mixture by using a spectrophotometer individually. The experiment was repeated three times and the mean value was taken for analysis of data.

Effect of time on Ca(II)-EDTA complex formation

To observe the effectiveness of EDTA solution, the absorbance was measured with the help of a spectrophotometer (UV-Vis spectrophotometer, ELICO SL 177) from 0-60 minutes at an interval of five minutes with 20 mg/L solutions at pH 1.5.

From the metal concentration measured before and after adsorption (c_i and c_e, respectively) and dry weight of adsorbent (W), the concentration of Ca (II) ions adsorbed in mg/L at equilibrium are computed by the formula,

$$q = \frac{c_i - c_e}{w} \times V \text{ (mg/g)} \text{......................} \text{(i)}$$

where, c_i and c_e are the concentration of Ca(II) ions in mg/L initially and at equilibrium respectively. V is the volume of the Ca(II) ions in the liter and W is the weight of adsorbent in gram.

Removal percentage is defined as the ratio of the decrease in metal ion concentration before and after adsorption to the initial concentration of metal in aqueous solutions.

$$\text{So, Percentage amount (A \%)} = \frac{c_i - c_e}{c_e} \times 100 \text{................} \text{(ii)}$$

$$\text{At equilibrium, } D = \frac{q_e}{c_e} \text{ ....} \text{(iii)}$$

where 'A%' is the adsorbed metal percentage from the solution and 'D' is the distribution factor of the adsorption in (L/g).

Results and Discussion

The sorption of Ca(II) ions in an aqueous solution were examined by optimizing various physicochemical parameters such as pH, adsorbate concentration, contact time, adsorbent dose. The absorbance of a blue-pink colored complex of Ca(II)–EDTA complex in H_2SO_4 solution at a maximum wavelength of 520 nm, i.e., λ_{max} is 520 nm.

Effect of pH for adsorption of Ca(II) ion onto CWH

The pH of the solution is the most important parameter in the removal of the metal ion by adsorption. The removal of Ca(II) ion was found to be decreased with increasing pH. The maximum removal value was found to be 1.5 and this is an optimum pH for CWH as shown in Figure 1.

Effect of time on Ca(II)-EDTA complex formation

By measuring the absorbance at the interval of five minutes, it was found that the value of absorbance was decreasing slightly (Figure 2). It was seen that from 0–20 minutes, the value of absorbance was found almost constant as shown in Figure 2. So, we measured the value of absorbance within 20 minutes by using EDTA solution.

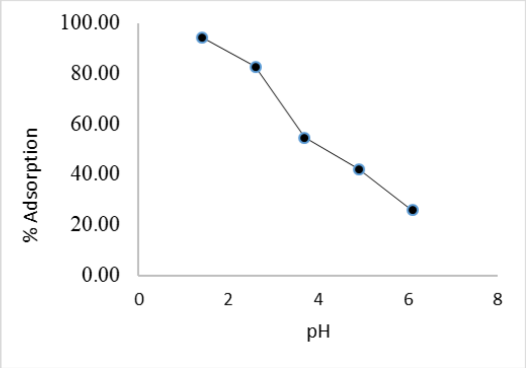


***Figure 1**: Effect of pH on Ca(II) ion adsorption by CWH*

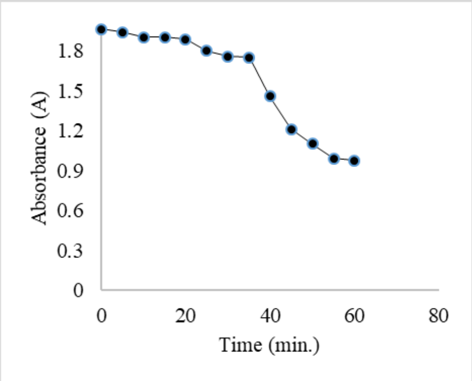


***Figure 2**: Effect of time on Ca(II) EDTA complex formation*

Effect of contact time for adsorption of Ca(II) ion onto CWH

Figure 3 depicts the study of adsorption which was carried out in measuring the effect of contact time on the batch adsorption of 50 mg/L metal solution with 25 mg adsorbent for different time intervals ranging from 10 minutes to 360 minutes. The required time equilibrium for adsorption of Ca(II) ion onto CWH was found to be 250 minutes. The percentage of adsorption is gradually increased at the start and then remains almost constant after attaining a time equilibrium of 250 minutes due to the availability of a large surface area of adsorbent initially but later active sites of adsorbent get saturated [22].

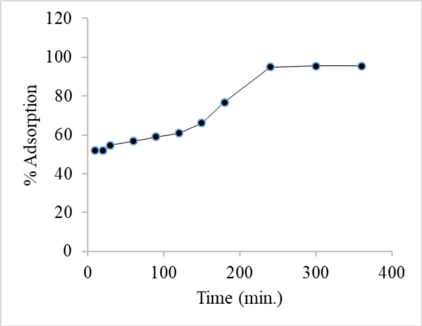


***Figure 3**: Effect of contact time for the adsorption percentage of Ca(II) ions onto CWH*

Effect of Ca(II) ion concentration

Figure 4 shows the effect of concentration in the adsorption of Ca(II) ions onto CWH. The adsorption increases from a lower concentration to a higher concentration ranging from 5 mg/L to 200 mg/L, the adsorbed amount increases from 43.2 mg/g to 319.75 mg/g. These data show that the adsorption is increased on increasing the amount of adsorbent.

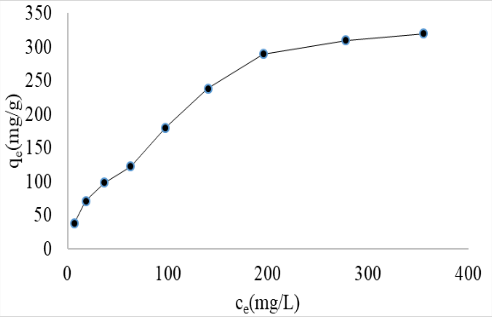


***Figure 4**: Effect of concentration in the adsorption of Ca(II) ions onto CWH*

Batch isotherm studies

Adsorption of Ca(II) ions onto CWH gives a linear relationship with Freundlich and Langmuir isotherms. Freundlich adsorption isotherm is more applicable due to having a greater correlation coefficient ($R^2 = 0.98$) than Langmuir adsorption isotherm ($R^2 = 0.946$). The Freundlich and Langmuir plots for the adsorption of Ca(II) ions onto CWH are shown in Figure 5 and Figure 6, respectively. The limited form of Freundlich isotherm is given by the following equation.

$$q_c = Kc_e^{1/n} \text{.........................} \quad \text{(iv)}$$

In logarithmic form,

$$\text{Log } q_c = \log K + 1/n \log c_e \text{..............} \quad \text{(v)}$$

Where q_c is the amount adsorbed per unit mass of adsorbent (mg/g), c_e is the equilibrium concentration of the adsorbent mg/L, K and n are Freundlich equilibrium coefficients, which are considered to be relative indicators of adsorption capacity and adsorption intensity [24]. The better fitting of Freundlich model suggested the formation of multilayer coverage of Ca(II) ions on the surface of CWH.

Batch kinetic studies

The kinetic data for the adsorption of Ca(II) ion onto CWH was studied by using pseudo-second-order and second-order kinetics model. From these results, the pseudo-second-order model was found to be more suitable to describe adsorption kinetics than the second-order kinetics [20]. The pseudo-second-order kinetics is suitable for Ca(II) adsorption due to the higher value of R^2 (0.99), indicating the adsorption is chemisorption. The plots of both second-order and pseudo-second-order models are shown in Figure 7 and Figure 8, respectively.

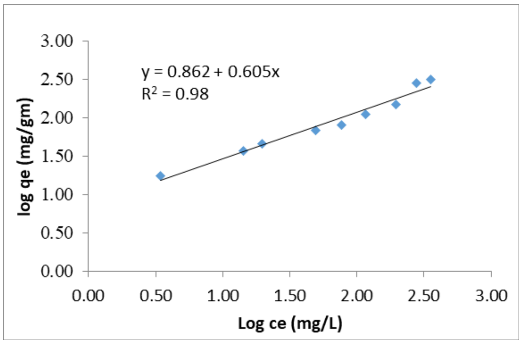


***Figure 5**: Freundlich isotherm plot for the adsorption of Ca(II) ion onto CWH*

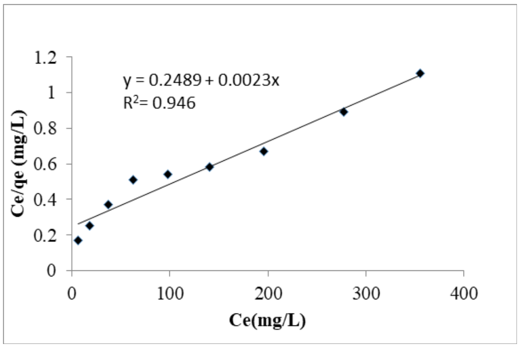


***Figure 6**: Langmuir isotherm plot for the adsorption of Ca(II) ion onto CWH*

Determination of adsorption efficiency

Determination of Ca(II) ion concentration was performed by EDTA titration. The adsorption efficiencies of activated carbon for each quantity of Ca(II) solution was calculated as follows:

Efficiency = (initial concentration – collected concentration) × 100 / initial concentration….. (vi)

The graph of adsorption efficiencies and collected volumes of Ca(II) ion solution is plotted in Figure 9.

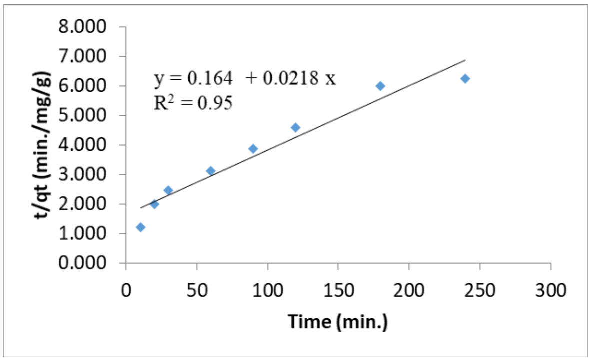


***Figure 7**: Second order kinetic plot for the adsorption of Ca^{2+} ion onto CWH*

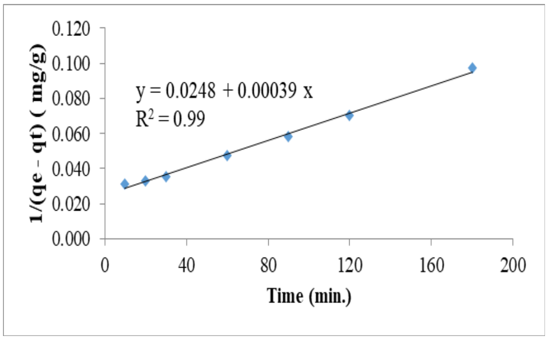


***Figure 8**: Pseudo-second order kinetic plot for the adsorption of Ca(II) ion onto CWH*

It was found that the highest adsorption efficiency of activated carbon for Ca(II) ion was about 91.8%.

The comparison of the isotherm and kinetic parameters for different metal ions using CWH as adsorbent is mentioned in Table 1.

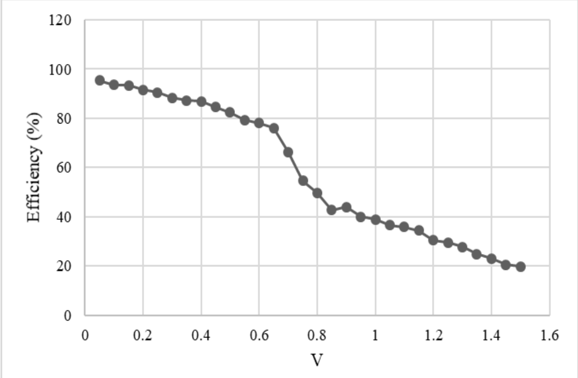


***Figure 9:** Efficiency of the column (in %) concerning the volumes of the collected solution of Ca(II) (in L)*

SEM and EDX studies

SEM image and EDX spectra (JEOL JSM – 6700F) were carried out to study the morphology and elemental composition of the adsorbent. Figure 10 depicts the SEM micrograph of charred water hyacinth.

The irregular and very rough surfaces could be observed in chemically modified water hyacinth

***Table 1**: Comparison of isotherm and kinetic parameters for different metal ions*

Metal ions using CWH	Isotherm	Kinetics	Q_{max} (mg/g)	pH	Efficiency (%)	References
Zn(II)	Langmuir	Pseudo 2nd order		6	79.6	[32]
Cu(II)	Langmuir	Pseudo 2nd order		6	92	[32]
Cr(VI)	Langmuir	Pseudo 2nd order	101.01	2		[33]
Cd(II)	Langmuir	Pseudo 2nd order	104.16	6	70	[19]
Ca (II)	Freundlich	Pseudo 2nd order	319.75	1.5	91.8	This study

which confirms that the hydrolysis reaction occurs on the surface of water hyacinth by the action of acidified $ZnCl_2$ solution. The irregular and rough surfaces were developed by $ZnCl_2$ due to the cleavage of polymeric networks of water hyacinth and converted it into small particles with large volume or surface area.

Figures 11(a) and 11(b) show the EDX spectra of adsorbent before and after Ca(II) ion adsorption, respectively. The presence of a peak for calcium ion is seen in EDX spectra only after adsorption, which confirms the good adsorption of calcium.

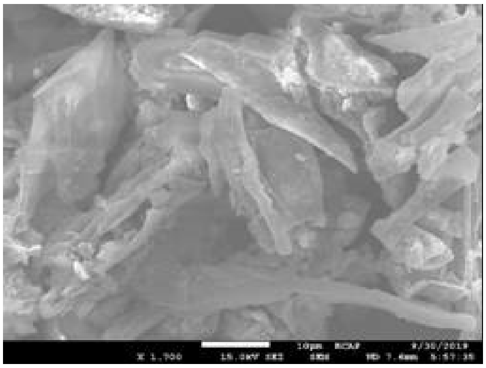


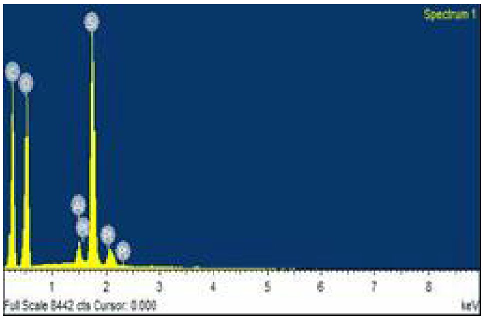
***Figure 10**: SEM micrograph of charred water hyacinth*

***Table 2(a):** Elemental composition of CWH before Ca(II) ion adsorption*

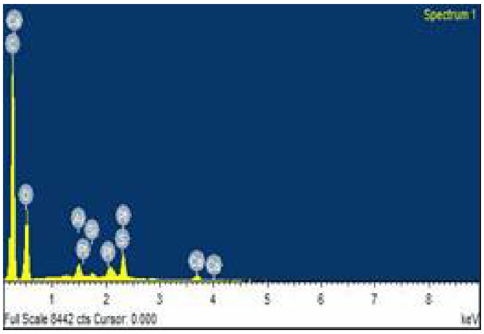
Elements	Weight %	Atomic %
C K	45.66	57.29
O K	37.28	35.21
Al K	1.06	0.59
Si K	12.36	6.63
Pt K	3.64	0.28
Total	**100.00**	**100**

***Table 2(b):** Elemental composition of CWH after Ca(II) ion adsorption*

Elements	Weight %	Atomic %
C K	61.17	69.14
O K	30.19	26.36
Al K	1.02	0.53
Si K	1.38	0.40
Ca K	2.93	1.69
Pt L	1.52	0.88
Total	100.00	100



(a)



(b)

***Figure 11:** EDX spectra of adsorbent (a) before and (b) after Ca(II) ion adsorption*

Table 2(a) and Table 2(b) depict the elemental composition of CWH before and after Ca(II) ion adsorption, respectively obtained from EDX analysis. As given in Table 2(a), only main elements (C and O) along with some other elements (Al, Si, and Pt) are observed, but the presence of Ca(II) ion as shown in Table 2(b) suggests that it is successfully adsorbed by CWH.

Conclusion

The low-cost adsorbent, water hyacinth, can be used as a new type of adsorbent for the removal of Ca(II) ions by the adsorption process. The optimum pH for the adsorption of Ca(II) ions is 1.5 and the Freundlich adsorption isotherm model with correlation coefficient R^2 of 0.98 was more applicable than the Langmuir isotherm model (R^2=0.946). The maximum adsorption capacity of Ca(II) ions onto CWH is found to be 319.75 mg/g. The required time equilibrium for the adsorption of Ca(II) ions onto CWH was found to be 250 minutes. The experimental data were better described by the pseudo-second-order kinetics model. The adsorbed concentration of Ca(II) ion was calculated by EDTA titration and efficiency was found to be 91.8%. Due to the presence of a rough surface, verified from the SEM image, charred water hyacinth has greater efficiency of the adsorption capacity. The EDX spectra indicate the successful adsorption of the Ca(II) ions.

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