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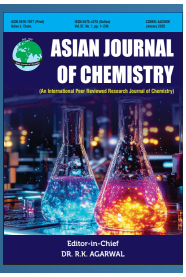

Calamondin Peel Waste-Derived Activated Carbon as a Biosorbent for Heavy Metal Removal from Aqueous Solutions

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This research focused on utilizing agricultural waste, calamondin (*Citrus madurensis*) peel, to produce low-cost calamondin peel-based activated carbon (CPAC), initially for Pb(II) ion removal from simulated water. CPAC, prepared by carbonizing chemically treated peel waste at 400 ºC for 120 min, achieved 99% Pb(II) removal under optimized conditions: 5 ppm initial Pb(II) concentration, 0.03 g adsorbent dosage, 20 min shaking time at pH 7. Isotherm studies confirmed chemisorption, with a maximum adsorption capacity of 10.63 mg/g. Furthermore, CPAC demonstrated a removal efficiency of 31.01% to 49.29% for Cd, Cr, Cu, Mn and Zn in water samples collected from the Kelani river, Sri Lanka. These findings emphasize the viability of CPAC as a cost-effective, environmentally sustainable adsorbent for the remediation of heavy metal-laden water, thereby advancing sustainable waste management and environmental remediation.

Keywords: Activated carbon, Adsorption, Isotherms, Heavy metal removal.

INTRODUCTION

With the rapid growth of industries such as electroplating, batteries, mining operations and fertilizers, direct or indirect discharge of wastewater containing heavy metals into the environment has significantly increased [1]. Therefore, considerable amounts of heavy metals have been detected in industrial waste streams [2] and several heavy metal removal techniques have been developed.

Among the contaminant removal techniques, adsorption has widely been used for the removal of pollutants from wastewater, due to the lower installation and operation cost and reversible nature of the process *i.e.*, ability to regenerate the adsorbent using a suitable desorption method [3,4]. Among several adsorbents including zeolites, clays, minerals and metal oxides, adsorption onto activated carbon has been identified as a more efficient and cost-effective method [5]. Its effectiveness originates mainly from its extensive surface area and significant micropore and mesopore capacities. Numerous studies [6-8] have focused on the economical removal of contaminants from aqueous solutions using activated carbon. However, as the price of commercially available coal-based activated carbon has increased due to the depletion of these natural sources [2], new concerns have emerged about the development and use of low-cost bio-based sorbents for wastewater purification [9], mainly because of the high availability, sustainability, efficiency and economic feasibility of these materials [10].

Several biomass and agricultural wastes, including orange peels, banana peels, potato peels, mango peels, rice husks and sugarcane bagasse, *etc.* have been utilized in the preparation of activated carbon to eliminate heavy metals from aqueous solutions [11]. Food processing bio-waste, such as calamondin peels, has little economic value other than using it for animal food, fuel and manure. Because of the higher yield and relatively high consumption, a considerable amount of fruit peel is produced and accumulates as biomass waste, resulting in disposal issues. The presence of organic compounds such as cellulose, hemicellulose and pectin in the calamondin peels [12], makes it possible to obtain environmentally beneficial biochar and activated carbon through pyrolysis. However, the effect of the physico-chemical properties of the activated carbon derived from calamondin peels on eliminating heavy metals in wastewater [13] has not been extensively investigated. Therefore, the present study focused on the determination of the potential of utilizing agricultural waste, peel waste of *Citrus madurensis* (calamondin) as precursors to produce low-cost activated carbon for the elimination of Pb(II) ions from aqueous solutions. The objective of this study was to prepare, characterize and evaluate the Pb(II) removal efficiency of activated carbon derived from *Citrus madurensis* peel waste, calamondin peel-based activated carbon (CPAC) and to evaluate its heavy metal removal efficiency with wastewaters collected from several locations in the Kelani river of Sri Lanka.

EXPERIMENTAL

All analytical-grade chemicals and reagents were sourced from Sigma-Aldrich, USA, Research Lab (India) or Fluka (Switzerland). The experiments were conducted using deionized water and calamondin fruits were purchased from local markets in Kiribathboda and Colombo, Sri Lanka.

Preparation of activated carbon from calamondin peel waste at different carbonization times: Washed and cleaned calamondin peels were sun-dried for 3 days and these samples (10.00 g) were chemically activated with H_3PO_4 (0.01 M) and carbonized at 400 ºC for 60-120 min (60 min (AC60), 90 min (AC90) and 120 min (AC120). The activated carbon yield percentages were calculated using eqn. 1:

$$Y\ (\%) = \frac{m}{M} \times 100 \tag{1}$$

where Y is the yield percentage of CPAC; m is the dry weight of CPAC (g) and M is the dry weight (g) before activation.

Determination of effect of carbonization time on the Pb(II) removal percentage by CPAC: To evaluate the Pb(II) removal percentage of CPAC prepared under different carbonization times, a Pb(II) standard solution (10.00 mg/L) was added separately into CPAC samples (0.02 g, AC60, AC90, AC120) and stirred for 10 min at room temperature and pH 7. The pH of the solutions was adjusted to pH 7 by adding desired volumes of NaOH or HCl (0.1 M). The solutions were filtered and the filtrate was transferred into a separatory funnel. Next, distilled water (25 mL) and ammonical-sulphite-cyanide solution (60 mL) were added to the separatory funnel. Dithizone solution (50 ppm, 25 mL) in dichloromethane as the complexing agent was added to the same separatory funnel, shaken for 2-3 min and allowed the mixture to separate. After separation, the organic layers were collected separately into clean dry conical flasks through dry filter papers and the residual lead (Pb^{2+}) concentrations in the filtrates were determined from a constructed calibration curve by measuring the absorbance using UV-vis spectrometer (Orion AquaMate 8000) at a wavelength of 515 nm and the Pb(II) removal percentage was calculated from eqn. 2:

$$\text{Pb(II) removal}\ (\%) = \frac{C_i - C_e}{C_i} \times 100 \tag{2}$$

where C_i is the initial Pb(II) concentration (mg/L) and C_e is the final Pb(II) concentration (mg/L) after adsorption onto the CPAC. The CPAC sample with the highest Pb(II) removal percentage was characterized by scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDAX), Fourier transform infrared spectroscopy (FT-IR) and proximate analysis for moisture, volatile matter, ash and fixed carbon contents of both raw peel powder and CPAC.

Characterization: The surface morphologies of the raw peel powder, prepared CPAC and lead adsorbed CPAC were determined by scanning electron microscopy (SEM) technique using a fully computer-controlled scanning electron microscope (Zeiss-Evo-18 Research^{TM}). EDAX was used to determine the elemental composition of the raw peel powder, prepared CPAC and lead-adsorbed CPAC. FTIR spectra of raw peel powder and prepared CPAC with the highest lead removal percentage were obtained from a Bruker Vertex 80 spectrometer using the Attenuated Total Reflectance (ATR) method.

Proximate analysis: Moisture, ash, volatile matter and fixed carbon contents in CPAC and raw peels were determined using ASTM standard methods [14].

Batch adsorption studies: Batch adsorption studies were conducted using the CPAC prepared by carbonizing raw material at 400 ± 30 ºC for 60 min.

Effect of initial Pb(II) concentration on adsorption of Pb(II) onto CPAC: Pb(II) solutions with different initial concentrations (1 mg/L, 2 mg/L, 3 mg/L, 4 mg/L and 5 mg/L) were added to 5 containers with CPAC (0.02 g) separately and shaken at room temperature (30 ± 2 ºC) for 60 min at pH 7. The filtrates were collected and analyzed by atomic absorption spectrophotometer (AAS) to determine the residual Pb^{2+} concentration.

Effect of initial activated carbon dosage on Pb(II) removal by CPAC: CPAC dosages (0.01 g, 0.02 g, 0.03 g, 0.04 g and 0.05 g) were separately introduced into clean and dry polypropylene containers and a volume of Pb(II) solution (25 mL, 5 mg/L) was introduced. The samples were shaken for 1 h at pH 7 and at room temperature. Above mentioned dosages were used for the preparation of control samples with deionized water (25 mL) and a similar experiment was carried out simultaneously. After shaking, all the samples were filtered and the residual Pb(II) concentration in the filtrates was determined using AAS.

Effect of shaking time: CPAC samples (0.03 g) were placed in clean and dried polypropylene containers of similar size. Then, 25 mL of Pb(II) solution (5 mg/L) was introduced into each of the containers with CPAC and these samples were shaken separately at different time intervals (20 min, 30 min, 60 min, 90 min, 120 min) at pH 7 and at room temperature (30 ± 2 ºC). After shaking, all the samples were filtered and analyzed to determine the residual Pb(II) concentrations in the filtrates using AAS.

Effect of the initial pH: The CPAC samples (0.03 g) were placed into clean and dry polypropylene containers and Pb(II) solutions (5 mg/L, 25 mL) with adjusted pH (pH 2, pH 3, pH 5, pH 7, pH 9) were introduced into the containers with CPAC. These samples were shaken for 20 min at room temperature (30 ± 2 ºC) and the filtrates were analyzed by AAS.

Isotherm studies: CPAC was taken (0.03 g) into clean and dry polypropylene containers and Pb(II) solutions with different concentrations (1 mg/L, 2 mg/L, 3 mg/L, 4 mg/L, 5 mg/L) were added separately into them. Next, the samples were shaken for 40 min to achieve the equilibrium at room temperature (30 ± 2 ºC) and at pH 7. Then the filtrates were analyzed using AAS and the collected data were used to calculate the residual Pb(II) concentration [15].

Wastewater samples: Wastewater samples from seven different locations of Kelani river (Table-1) were collected and stored in a cold box and transported to the laboratory within 24 h. Next, 25 mL of each of these samples was transferred into clean and dry polypropylene bottles and their initial pH, conductivity (EC) and total dissolved solids (TDS) were measured. These samples were filtered and analyzed for initial heavy metal ion concentrations (Cd, Cr, Mn, Cu, Zn) using the inductive coupled plasma-mass spectrophotometer (ICP-MS, Agilent 7800). Then, CPAC (0.02 g) was added to the wastewater samples (25 mL) collected from seven different locations of Kelani river. Each sample was shaken for 40 min, filtered and then the residual metal ion concentrations were determined by the ICP-MS.

TABLE-1
DETAILS OF SAMPLING LOCATIONS

Sampling location	Sampling points	GPS coordinates
Pugoda Ferry	01	6′98″00° N, 80′12″24° E
Hanwella Bridge	02	6′54″36° N, 80′05″00° E
Kaduwela Bridge	03	6′94″12° N, 79′98″51° E
Thotalanga Bridge	04	6′96″43° N, 79′87″93° E
Biyagama Junction	05	6′94″16° N, 79′98″72° E
Raggahawatte Ela (Tributary)	06	6′56″21° N, 79′58″09° E
Ma-Oya (Tributary)	07	7′19″45° N, 80′07″23° E

Statistical analysis: All experiments were conducted in duplicate and the results are presented as the mean ± standard deviation.

RESULTS AND DISCUSSION

The effectiveness of removing Pb(II) using prepared CPAC over different carbonization periods was assessed through a spectrophotometric technique. The results suggested that the yield % of CPAC depends on the carbonization time and the maximum yield % was obtained when CPAC prepared at the carbonization time of 120 min (Fig. 1) and the highest Pb(II) removal % (81.73%) was detected when the raw material was carbonized for 120 min (Fig. 2).

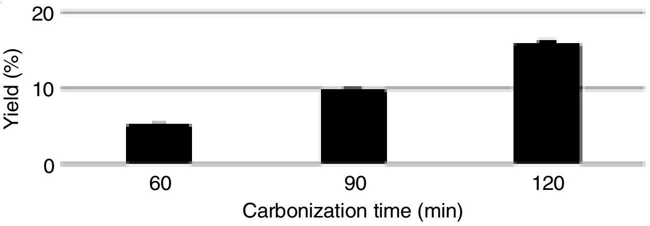

Fig. 1. Effect of carbonization time (min) on yield% of AC

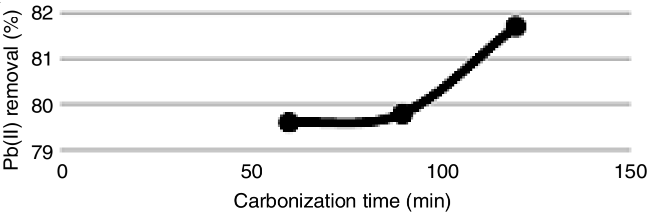

Fig. 2. Effect of carbonization time (min) on the Pb(II) removal %

Proximate analysis of CPAC: For the raw material to be considered a precursor for activated carbon, it must include a substantial amount of volatile matter. High-temperature treatment can release volatile materials while improving the porosity and carbon levels of the material. The proximate composition of raw material and the CPAC is shown in Table-2. These results suggest that when the raw material is activated, the volatile content decreases from 31.87% to 11.65%, possibly due to the release of volatile matter through chemical activation, while the carbon content increases from 10.67% to 31.03%. These results suggest the suitability of CPAC as an adsorbent material.

TABLE-2
PROXIMATE ANALYSIS RESULTS

Test	Compositions of each sample (%)	
	Raw sample	Activated carbon
Moisture content	12.98	8.71
Volatile matter	31.87	11.65
Ash content	44.48	48.61
Fixed carbon content	10.67	31.03

FTIR spectroscopy: The FTIR spectra of the raw material and CPAC were obtained within the range of 3400-875 cm^{-1} (Fig. 3). Upon carbonization along with activation, it is clear that the broad and intense peaks in the spectrum of the raw peel powder attributed to the -OH stretching vibration at 3500-3200 cm^{-1} -CH, -C=O and C-OH stretching vibrations at 2930 $\mathrm{cm}^{-1,}$ 1625 cm^{-1} and 1022 cm^{-1} respectively were either reduced their intensities, disappeared or shifted [16].

SEM and EDAX: As Fig. 4 depicts, raw peel powder (a) has irregular cracks and voids, while the prepared activated carbon (b) and the Pb(II) adsorbed activated carbon (c) have more regular pores with an average diameter of 2.0 µm. Therefore, the activation process results in a more regular honeycomb-shaped porous structure, with a higher surface area, leading to many adsorption sites.

The elemental compositions of the materials determined by EDAX are given in Table-3. A peak related to Pb(II) was observed in the EDAX spectrum of Pb(II) adsorbed activated carbon, confirming the adsorption of Pb(II) onto the prepared CPAC under the selected experimental conditions.

TABLE-3
ELEMENTAL COMPOSITION OF RAW CALAMONDIN PEEL POWDER, CPAC AND Pb(II) ADSORBED CPAC

Sample	Weight (%)				
	C	O	Ca	Mg	Pb
Raw calamondin peel powder	57.39	39.39	2.98	0.29	–
Activated carbon	69.58	19.81	9.69	0.92	–
Pb(II) adsorbed AC	76.69	15.33	7.19	0.57	0.23

Batch adsorption studies: Batch studies were carried out using a synthetic Pb(II) solution and the effect of the initial Pb(II) concentration, CPAC dosage, shaking time and solution pH on the Pb(II) removal efficiency by CPAC were evaluated. First, the percent removal of Pb(II) was determined by varying the initial Pb(II) concentration (Fig. 5a) within a range of 1-5 mg/L and the results showed that when the solution concentration was increased from 1-5 mg/L, the metal removal percen-

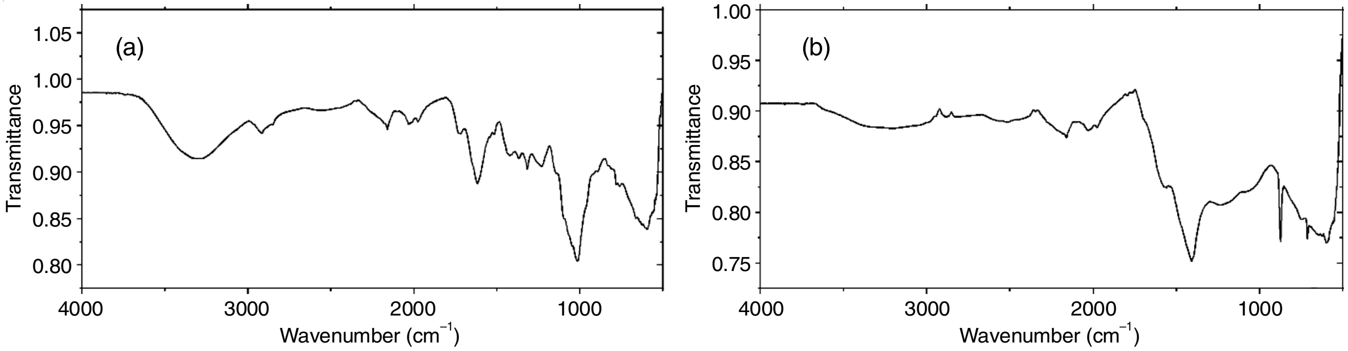

Fig. 3. FTIR spectra of (a) raw calamondin peel powder and (b) activated carbon derived from calamondin peels

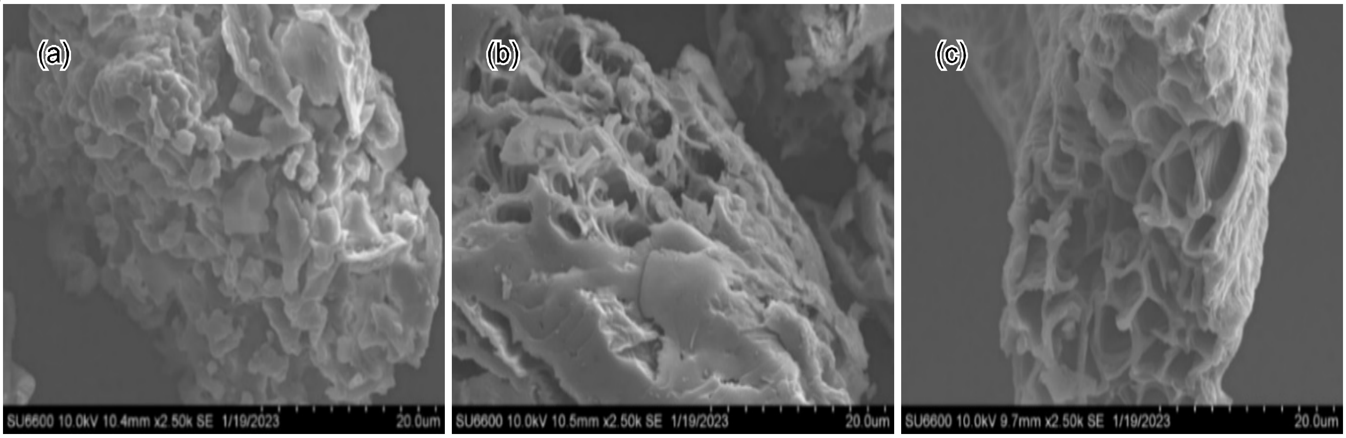

Fig. 4. SEM images of (a) raw peel powder (b) CPAC (c) and Pb adsorbed CPAC (magnification = x2.50k)

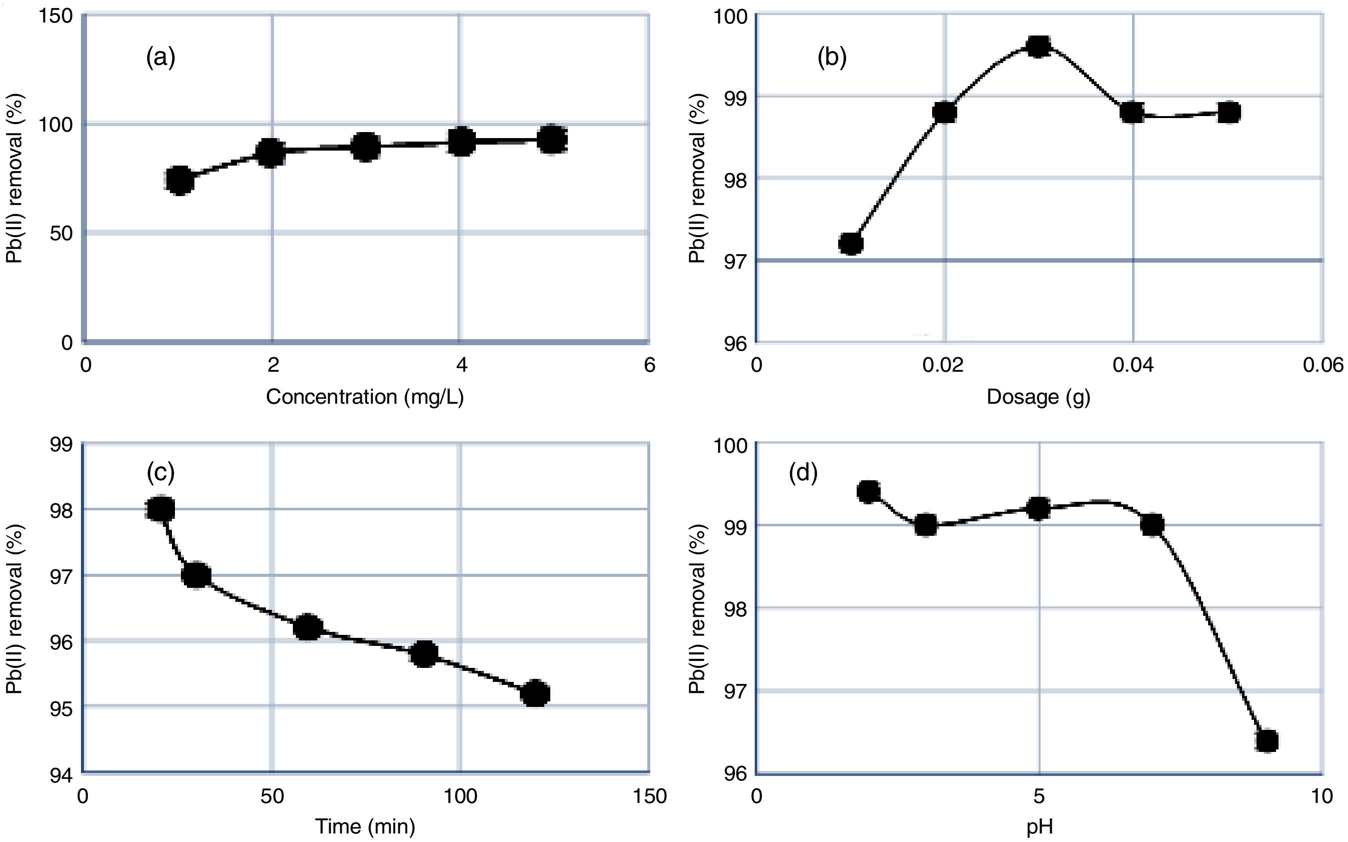

Fig. 5. Batch adsorption study under the parameters of (a) initial Pb(II) concentration (b) CPAC dosage (c) shaking time and (d) initial solution pH

tage by the CPAC also increased. This trend might be attributed to the higher availability of adsorption sites on CPAC at lower Pb(II) concentrations and the saturation of available sites with metal ions with the increase of the concentration. To evaluate the effect of CPAC dosage, CPAC ranging from 0.01 g to 0.05 g was introduced to Pb(II) solutions at a fixed concentration of 5 mg/L at pH 7 with a shaking time of 60 min (Fig. 5b). When the dosage of CPAC was increased from 0.01 g to 0.05 g, the metal removal percentage also increased and the maximum removal was observed at a CPAC dosage of 0.03 g, which was 99.60%. After that the Pb(II) removal suddenly decreased and became almost constant, which may be due to the aggregation of CPAC particles with the increasing CPAC dosage while reducing the number of available sorption sites to adsorb Pb^{2+} ions from the solution [17]. The effect of Pb(II) removal over the shaking time is graphically represented in Fig. 5c and among the selected contact times, the maximum Pb(II) removal (98%) was observed after 20 min of shaking. When the shaking time increased, the percentage removal of Pb(II) decreased, possibly due to the desorption effect at longer shaking times. Since the solution pH significantly governs the adsorption process of metal ions, the effect of the pH solution on Pb(II) adsorption onto CPAC was evaluated by varying the pH from 2-9 (Fig. 5d). Accordingly, the maximum Pb(II) removal percentage was observed within the range of pH 2-pH 7 and with the increase of solution pH, the Pb(II) removal percentage decreased and this might be due to the precipitation of Pb(II) ions as hydroxides [18]. The batch adsorption studies suggested that the highest Pb(II) removal percentage (99%) occurred at an initial concentration of Pb(II) 5 ppm, an adsorbent dosage of 0.03 g and 20 min shaking time at pH 7 at room temperature (30 ± 2 ºC).

Isotherm studies: Adsorption isotherm models describe the interaction between an adsorbate and an adsorbent during the equilibrium phase. In this study, the linearized forms of the Langmuir and Freundlich isotherms were used. The Langmuir isotherm suggests that the interaction between the adsorbent and the adsorbate takes place as a monolayer form on the surface of the adsorbent. The linearized form of Langmuir equation is represented as follows [19]:

$$\frac{1}{q_e} = \frac{1}{bq_0C_e} + \frac{1}{q_0} \tag{3}$$

where q_e (mg/g) is the amount of adsorbate (mg) adsorbed per unit weight of adsorbent (g) at equilibrium; C_e is the equilibrium concentration of adsorbate; b is the adsorption coefficient and; q_0 is the maximum adsorbate absorbed onto the adsorbent. A plot of $1/q_e$ *vs.* $1/C_e$ gives $1/bq_0$ as the slope and $1/q_0$ as the intercept.

The Freundlich isotherm model can be applied to multilayer adsorption over a heterogeneous surface. The linearized Freundlich equation can be expressed as follows:

$$\log q_e = \log k + \frac{1}{n}\log C_e \tag{4}$$

where q_e is the amount of solute adsorbed per unit weight of adsorbent at equilibrium, C_e is the equilibrium concentration of adsorbate and k and n are the constants related to adsorption capacity and adsorption intensity, respectively.

Isotherm curves related to the Langmuir and Freundlich isotherm models obtained from the experimental data are given in Fig. 6a-b, respectively.

The Langmuir and Freundlich isotherm constants calculated from the experimental results are given in Table-4. Given that the adsorption data at the equilibrium phase, best fitted with the Langmuir adsorption isotherm compared to the Freundlich isotherm, it can be concluded that the Langmuir isotherm model can be used to describe the interaction between Pb(II) and the CPAC adsorbent in its equilibrium phase. This suggests that the available active sites of CPAC are homogeneous and that Pb(II) uptake occurs through chemisorption as a monolayer formation, indicating that there is no any interaction between the adsorbate molecules.

The dimensionless constant separation factor (R_L) related to the Langmuir isotherm plot can be utilized to determine if a

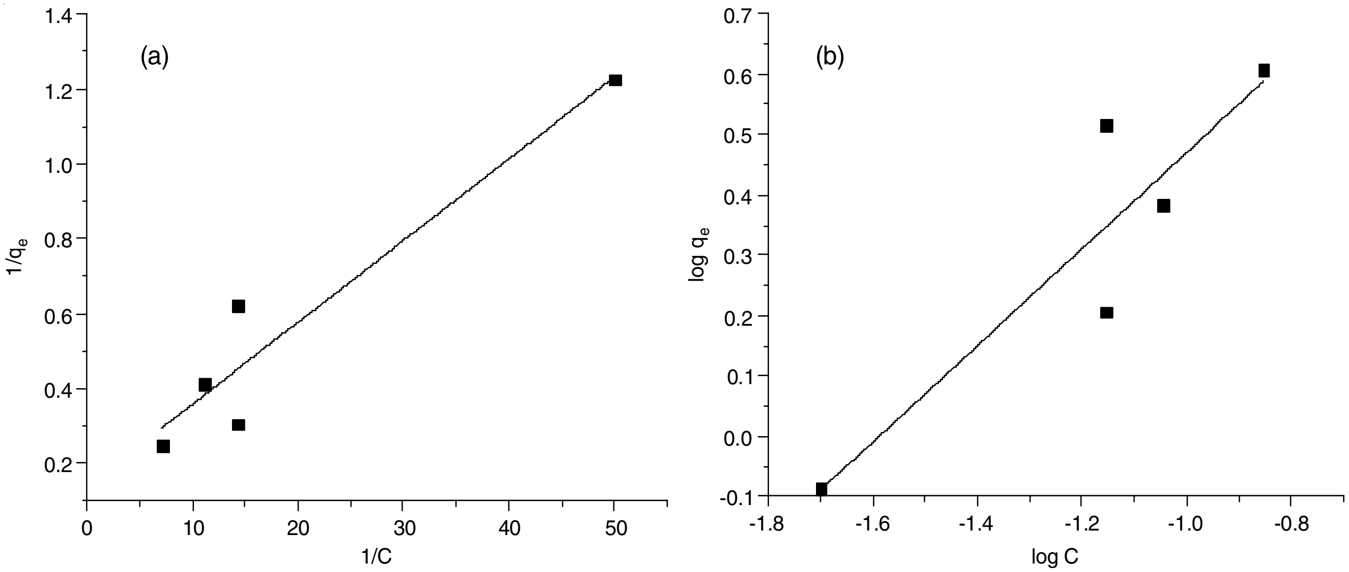

Fig. 6. (a) Langmuir adsorption isotherm of Pb(II) sorption onto CPAC, (b) Freundlich adsorption isotherm of Pb(II) sorption onto CPAC

TABLE-4
ISOTHERM PARAMETERS RELATED TO
THE Pb(II) ADSORPTION ONTO CPAC

Langmuir constants			Freundlich constants		
q_0 (mg/g)	b	R^2	k	n	R^2
10.63	6.36	0.91481	18.67	1.25	0.83259

sorption system is favourable or unfavourable in a batch adsorption process. The dimensionless constant separation factor (R_L) can be defined by the following equation:

$$R_L = \frac{1}{1+bC_i} \tag{5}$$

where C_i is the initial concentration (mg/L) of adsorbate; and b is the Langmuir equilibrium constant (L/mg).

The separation factor value (R_L) indicates that the Langmuir isotherm model type is irreversible (when $R_L = 0$), favourable ($0 < R_L < 1$), linear ($R_L = 1$) or unfavourable ($R_L > 1$) [20].

The maximum and minimum R_L values obtained for the initial Pb(II) concentrations ranging from 1-5 mg/L were 0.69 and 0.14, respectively. Since these values are between the 0-1 range, it can be concluded that the batch adsorption process is favourable for the total concentration range selected.

Applications: The initial pH, EC, TDS and the heavy metal removal efficiency of the prepared activated carbon on surface water samples collected from seven locations in the Kelani river were evaluated in this study. The analysis revealed that the pH (7.56-6.72), EC (349–46.2 µS/cm) and TDS (165.7–21.6 ppm) values are in the acceptable range of WHO and SLSI standards [21,22]. Fig. 7 shows the initial concentrations of the selected heavy metals (Cd, Cr, Mn, Cu, Zn) and their final concentrations after treatment of the water samples with CPAC. Pb(II) was not detected in any of the collected water samples.

The results revealed that the initial heavy metal concentrations of the water samples collected from the seven different locations were well below the permissible limits defined by SLSI and WHO [21,22]. The maximum average metal removal observed at 49.29% for Cd, while the minimum average was 31.01% for Zn following treatment with CPAC. The differences in heavy metal removal percentages by CPAC across various locations could be attributed to fluctuations in solution pH, initial metal ion concentrations, electrical conductivity, and total dissolved solids levels. It can be concluded that the CPAC performed well in Cd removal in a multi-ion solution.

Conclusion

In this work, the novel activated carbon was successfully derived from the low-cost and readily available agricultural waste of calamondin fruit peels. Upon carbonization and chemical activation, the novel adsorbent, calamondin peel-based activated carbon (CPAC) developed superior surface characteristics such as higher functionalized surface porosities as determined by the SEM/EDAX and FTIR data, resulting in higher metal removal efficiency. The highest Pb(II) removal percentage (99%) was observed with an initial Pb(II) concentration of 5 mg/L, an adsorbent dosage of 0.03 g, with a 20 min of shaking time at pH = 7 at room temperature (30 ± 2 °C). Furthermore, the isotherm studies revealed that the equilibrium data were

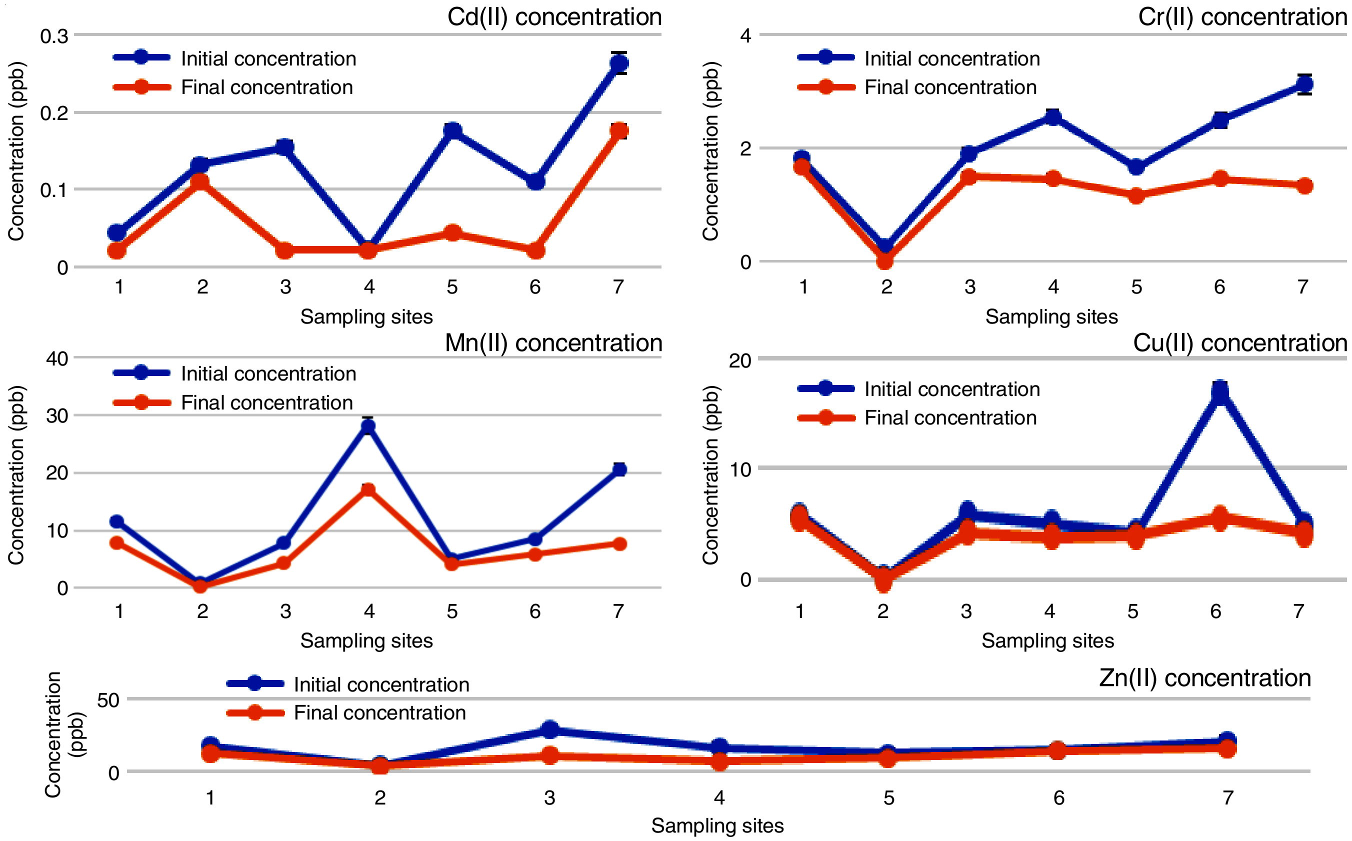

Fig. 7. Initial and final concentrations of the selected heavy metals

best fitted and can be presented using the Langmuir isotherm model, which provided a correlation coefficient of $R^2 = 0.91481$ and indicated the maximum adsorption capacity (q_0) of 10.63 mg g^{-1}, indicating that the Pb(II) adsorption onto the CPAC occurred *via* chemisorption. When the CPAC was used to determine the heavy metal removal efficiency of water samples collected from several locations in the Kelani river, the maximum average metal removal percentage (49.29%) was observed for Cd, while the least removal (31.01%) was observed for Zn. Also, CPAC could potentially eliminate all the selected heavy metals (Cd, Cr, Cu, Mn, Zn) from the collected water samples of the Kelani river. Thus, it can be suggested that the novel adsorbent, CPAC can be used as an efficient, cost-effective and eco-friendly adsorbent for the treatment of wastewater contaminated with the heavy metals.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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