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# Adsorption of cadmium from aqueous solution using Rooibos shoots as adsorbent

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The use of Rooibos shoots, a natural adsorbent, for cadmium removal from wastewater is proposed. The effects of initial pH, adsorbent dosage, contact time, and initial concentration were investigated in the batch adsorption mode. The optimum pH was found to be 5.5. Isotherm and kinetic data were modeled; the data fitted best to the Freundlich model, and, kinetically, the adsorption was of pseudo-second order as shown by the high $R^2$ value of 0.9928 along with close agreement between the experimental $q_e$ (13.9 mg $g^{-1}$) and calculated $q_e$ (14.24 mg $g^{-1}$) values. The studied biomass material was found to be effectively used for removal of cadmium from contaminated mine wastewater.

**Keywords:** Rooibos shoots; adsorption; cadmium; isotherm; kinetics

## 1. Introduction

Cadmium is one of the most hazardous trace metals due to its carcinogenicity and toxicity at low dose, together with good solubility and mobility in aqueous solutions (Waisberg et al. 2003). Cadmium may enter environmental waters through wastewater effluents from smelting, metal plating, and cadmium–nickel battery-producing industries. As it accumulates along the food chain, cadmium can cause human health hazards, leading to long-term effects such as renal dysfunction, hypertension, hepatic injury, and lung damage, and being teratogenic (Kazi et al. 2008). Toxicological studies have shown that short-term effects include nausea, vomiting, diarrhoea, and cramps (Kalkan et al. 2013). The maximum recommended concentration for drinking water established by the US Environmental Protection Agency is 0.01 mg $L^{-1}$ (Volesky 1990). Methods for removal of the metal ions from industrial wastewaters are therefore welcome, to avoid contamination of ground water utilized as a source of drinking water.

For removal of trace metals from aqueous solutions, approaches such as chemical precipitation, reverse osmosis, solvent extraction, electrodialysis, and membrane separation have been suggested (Al-Masri et al. 2010; Kumar et al. 2011; Guyo, Mhonyera, and Moyo 2014; Moyo et al. 2014). These methods have been proven effective, but they are time-consuming and usually produce large amounts of sludge that can add other environmental problems (Saleh and Gupta 2012; Daraei et al. 2013; Daraei et al. 2014). Therefore, adsorption technique using waste materials is gaining popularity due to its merits of low cost, simplicity, effectiveness, fast operation, and of being environmentally friendly (Ali and Gupta 2007; Gupta et al. 2009; Daraei et al. 2015; Heidari et al. 2013; Mittal,

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Thakur, and Mittal 2013; Yadav, Singh, and Sinha 2014; Mittal et al. 2014). In the last few years, several workers have tried different adsorbents such as slag (Gupta et al. 1997), bagasse fly ash (Gupta et al. 2003), rice straw (Ali et al. 2013), red mud (Gupta and Sharma 2002), teak leaves powder (Rao, Anand, and Venkateswarlu 2010a), maize tassel (Zvinowanda et al. 2009), orange peel (Gupta and Nayak 2012), and various other adsorbents (Mohan and Singh 2002; Abdel-Aty et al. 2013; Farhan, Al-Dujaili, and Awwad 2013; Naushad et al. 2014) for the removal of cadmium from wastewater.

The Rooibos bush is between 1 and 1.5 meter tall, shrubby, with thin needle-like leaves; the plant is a member of the legume family. It is indigenous to the Cedarberg region in the north-west of Cape Town, South Africa, where local communities have been using it for centuries to brew tea. On the bush, the leaves are green, but once processed they turn a deep red from the oxidization of the Rooibos leaves. The processing of the tea produces an agro-waste composed of coarse shoot materials, fine dust, and rejected grades in large quantities and which has no market value. It is important to extend the investigation on the potential of other agro-based plants and wastes to fill a much-needed niche in the overall carbon market which is extremely high. Therefore, the focus of the present study was to assess the potentiality of Rooibos shoots adsorbent for the removal of cadmium from aqueous solutions and wastewaters. The adsorbent was characterized using X-ray diffraction technique (XRD) and Brunauer–Emmett–Teller (BET) analysis. The effects of pH, adsorbent dosage, contact time, and initial cadmium concentration were investigated. The adsorption isotherm and sorption kinetics of cadmium onto Rooibos shoot powder are presented.

## 2. Materials and methods

### 2.1. Chemicals and instrumentation

$Cd(NO_3)_2$, NaOH, and $HNO_3$ from Sigma (Pretoria, South Africa) were of analytical grade and used as supplied. The surface area was estimated by BET analysis (A Tristar II 3020, Micrometrics BET from Norcross, GA, USA). XRD studies were carried out using an X-ray diffractometer (D8 Bruker, Germany) with Cu K$\alpha$ radiation at 40 kV and 40 mA. A flame atomic absorption spectrometer (AAS; Shimadzu AA-680, Japan) was used to measure the amount of cadmium. A pH meter (Crison 2001 micropH, Spain) was employed for measuring pH values in the aqueous phase.

### 2.2. Preparation of absorbent

The shoots, i.e. stem and leaves, were obtained from Rooibos (*Aspalathus linearis*) plants on fields in the Cedarberg region. The freshly collected shoots were hand-washed with tap water, followed by distilled water and then sun-dried. Drying at 60 °C in an oven for 72 h removed most of the moisture. The dried material was crushed to powder and the fine powder was further fractionated into different particle sizes. The size fraction ranging from 150 to 300 μm was used for adsorption studies.

### 2.3. Preparation of metal ion solution

A stock solution of 1 g $L^{-1}$ of cadmium ions was prepared by dissolving an appropriate amount of $Cd(NO_3)_2$ in distilled water. The concentrations used in the experiment were obtained by dilution of the stock solution.

### 2.4. Adsorption experiments

For studying removal of cadmium as a function of pH, eight 50 mL solutions of 100 mg $L^{-1}$ cadmium solution were pipetted into 100 mL Erlenmeyer flasks. The pH of the solutions was varied from 2.33 to 6.06. 0.4 g of Rooibos shoots was added in each of the container. The mixtures were agitated for 60 min at 150 rpm using the shaker. After equilibration, the samples were centrifuged for 3 min and filtered with a 0.45-μm membrane to separate the solid phase from the liquid phase after each batch experiment. The mixtures were acidified with two drops of concentrated $HNO_3$ and refrigerated at 4 °C until analysis by AAS.

For studying removal of cadmium as a function of dosage, ten 50 mL solutions of 100 mg $L^{-1}$ solution were pipetted into seven 100 mL bottles and varying masses from 0.4 to 2.2 g of Rooibos shoots were added. The mixtures were agitated for 60 min at 150 rpm using the shaker. After equilibration, the samples were centrifuged for 3 min and filtered with a 0.45-μm membrane to separate the solid phase from the liquid phase after each batch experiment. The mixtures were acidified with two drops of concentrated $HNO_3$ and refrigerated at 4 °C until analysis by AAS.

For studying removal of cadmium as a function of concentration, a standard solution of 1000 mg $L^{-1}$ was used to prepare different solutions in the range of 25−400 mg $L^{-1}$. Thereafter, 50 mL of the solutions were pipetted into seven 100 mL bottles and 0.4 g of the Rooibos shoots was added. The mixtures were then agitated for 60 min at 150 rpm using the shaker. After equilibration, the samples were centrifuged for 3 min and filtered with a 0.45-μm membrane to separate the solid phase from the liquid phase after each batch experiment. The mixtures were acidified with two drops of concentrated $HNO_3$ and refrigerated at 4 °C until analysis by AAS.

The experiment for the removal of cadmium as a function of time was carried out after pipetting eight 50 mL samples of 100 mg $L^{-1}$ cadmium solution into eight 100 mL bottles and followed by addition of 0.4 g of the Rooibos shoots. The mixtures were then agitated for 10, 20, 10, 30, 60, 90, 120, 150, and 180 min at 150 rpm using the shaker. After equilibration, the samples were centrifuged for 3 min and filtered with a 0.45-μm membrane to separate the solid phase from the liquid phase after each batch experiment. The mixtures were acidified with two drops of concentrated $HNO_3$ and refrigerated at 4 °C until analysis by AAS.

## 3. Results and discussion

### 3.1. Characterizations of Rooibos shoots

The measured BET surface area of the prepared shoots was 267 $m^2 g^{-1}$ and is higher than that of *Terminalia catappa* Linn leaf powder (8.4 $m^2$ $g^{-1}$) (Rao, Anand, and Venkateswarlu. 2010b) and teak leaves powder (6.35 $m^2$ $g^{-1}$) (Rao, Anand, and Venkateswarlu 2010a). The nitrogen adsorption–desorption isotherm at −196 °C of Rooibos shoots is shown in Figure 1. The shape of the isotherm indicates the mesoporous nature of the adsorbent prepared from the Rooibos plant. The adsorption hysteresis relates to the type IV isotherm since desorption curve lags behind adsorption curve. The pore size and pore volume of the adsorbent was 61.48 Å and 0.041 $cm^3$ $g^{-1}$, respectively.

The composition and physical properties of the adsorbent were determined using XRD. From the XRD pattern (Figure 2), it can be suggested that the adsorbent prepared from Rooibos plant consists of a mixture of amorphous and crystalline structure which

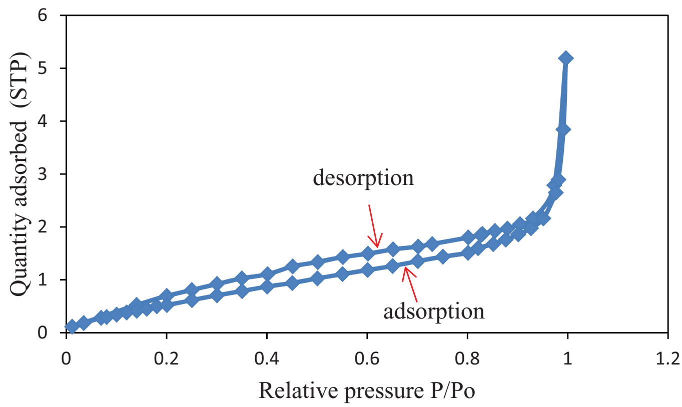


Figure 1. The nitrogen adsorption–desorption isotherm of the adsorbent prepared from Rooibos shoots.

gives a continuous function and had two partially, not well-defined resolved peaks at about $2\theta = 16.17°$ and $22.51°$, respectively (Moyo et al. 2013).

### *3.2. Optimization of sorption parameters*

The solution pH in adsorption studies mainly affects the surface charge, the degree of ionization, and the adsorbates species (Daraei et al. 2013; Guyo, Mhonyera, and Moyo 2014). The adsorption capacity of Rooibos shoots for cadmium from aqueous solution as a function pH was investigated (Figure 3).

The cadmium adsorption capacity increased very sharply with an increase in pH from 2.3 to 5.3. Under highly acidic conditions, the adsorption of cadmium was low, but it

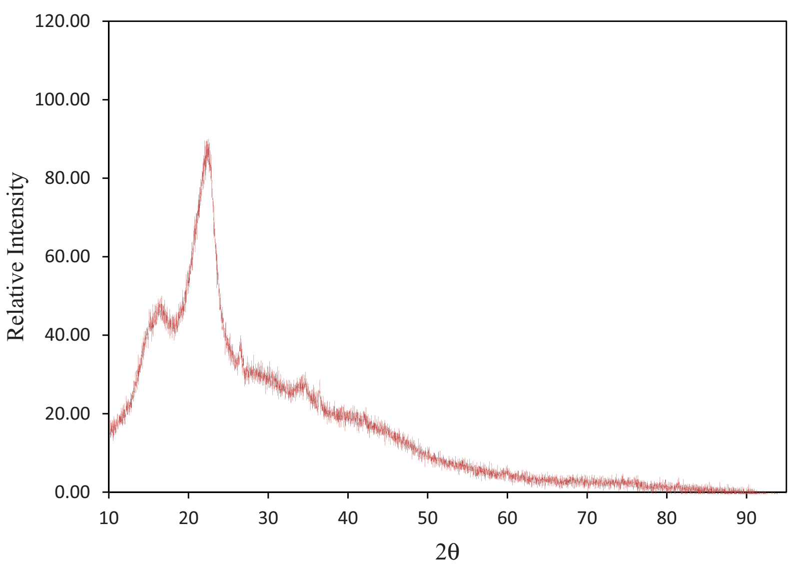


Figure 2. X-ray diffraction spectrum of Rooibos shoots.

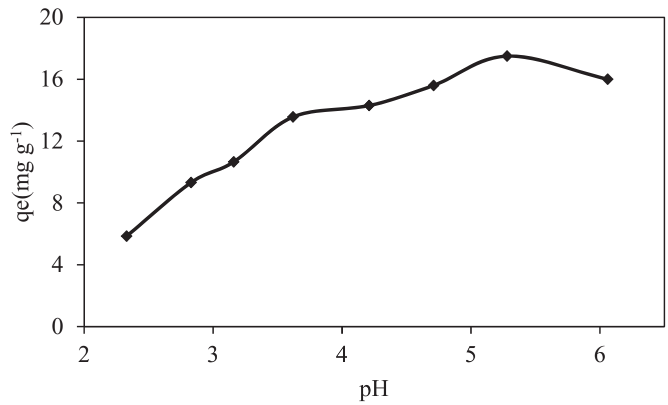


Figure 3. Effects of pH on the adsorption capacity of Rooibos shoots.

increased steadily as pH increases up to the maximum capacity of 59.76 mg $g^{-1}$. The lower biosorption capacity of the Rooibos shoots observed at highly acidic condition has been attributed to the competition that positively charged cadmium ions face from hydronium ($H_3O^+$) ions for the available adsorption sites. As the pH increases, the negative charge density on the adsorbent surface is exposed, thereby increasing the attraction of positively charged cadmium ions (Rao, Anand, and Venkateswarlu 2010a). Beyond the optimum pH of 5.3, the adsorbent shows a decrease in sorption attributed to cadmium hydrolyzing and precipitating instead of the adsorption process taking place (Singh, Rastogi, and Hasan 2005). Hence, further adsorption experiments were carried out at an optimum pH 5.3.

The dosage of an adsorbent strongly influences the extent of sorption. The adsorption capacity decreased from 12.51 to 3.11 mg $g^{-1}$ with an increase in Rooibos shoots adsorbent dosage (Figure 4). The adsorption capacity was low at high adsorbent dose due to less amount of cadmium per unit mass of adsorbent (Moyo et al. 2014). An adsorbent dose of 0.4 g was used in further experiments since it was sufficient for optimum adsorption of cadmium.

Figure 5 shows that as the concentration of cadmium increases, the adsorption capacity of the Rooibos shoots increased from 7.2 to 20.25 mg $g^{-1}$. An increase in adsorption

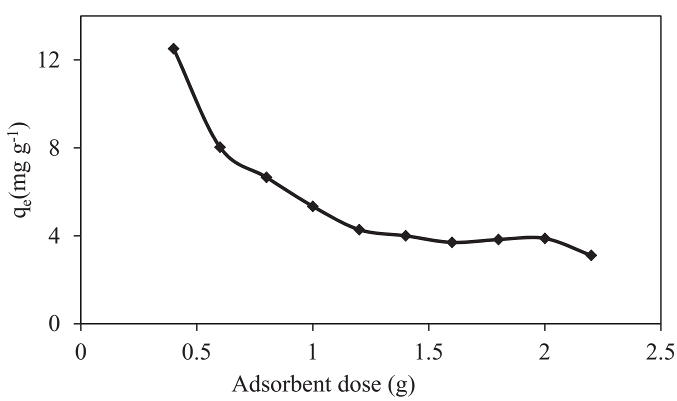


Figure 4. Effects of adsorbent dosage on the adsorption capacity of Rooibos shoots.

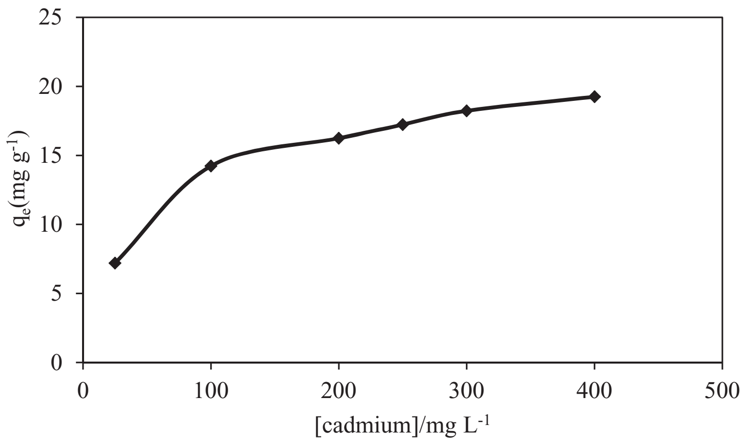


Figure 5. Effect of initial cadmium concentration on adsorption capacity of Rooibos shoots.

uptake might be attributed to increase in the driving forces such as the concentration gradient. On the other hand, the percentage adsorption of cadmium decreased from 82.40% to 70.55% (graph not shown) due to lack of sufficient surface area to accommodate much more cadmium available in the solution. Furthermore, an increase in the initial metal concentration imparts an increase in the mass transfer of the metal ion between the aqueous and solid phases, thereby increasing the loading capacity (Rao, Anand, and Venkateswarlu 2010a).

Adsorption of cadmium onto the Rooibos shoots with an increase in contact time was investigated (Figure 6). From the first minutes of interaction, there was an increase in adsorption capacity of the adsorbent for cadmium. The high sorption rate with increasing time is due to attraction of active functional groups towards cadmium, which leads to stronger surface binding. At 60 min, the system seems to have approached a steady state since no further change in adsorption was observed. As the surface sites of the Rooibos shoots become exhausted, the uptake rate is controlled by the rate with which the cadmium ions are transported from the exterior to the interior sites.

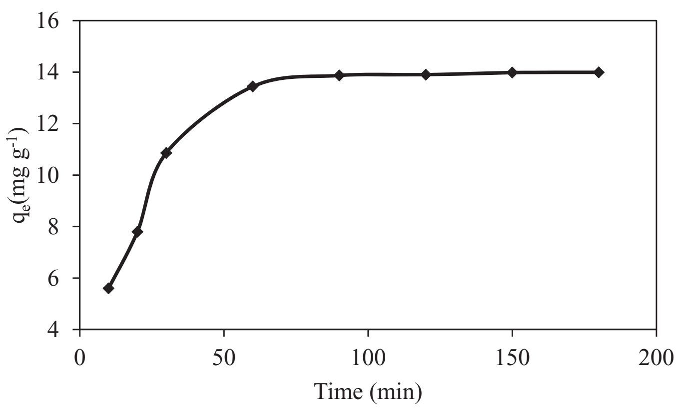


Figure 6. Effect of contact time on adsorption capacity of Rooibos shoots.

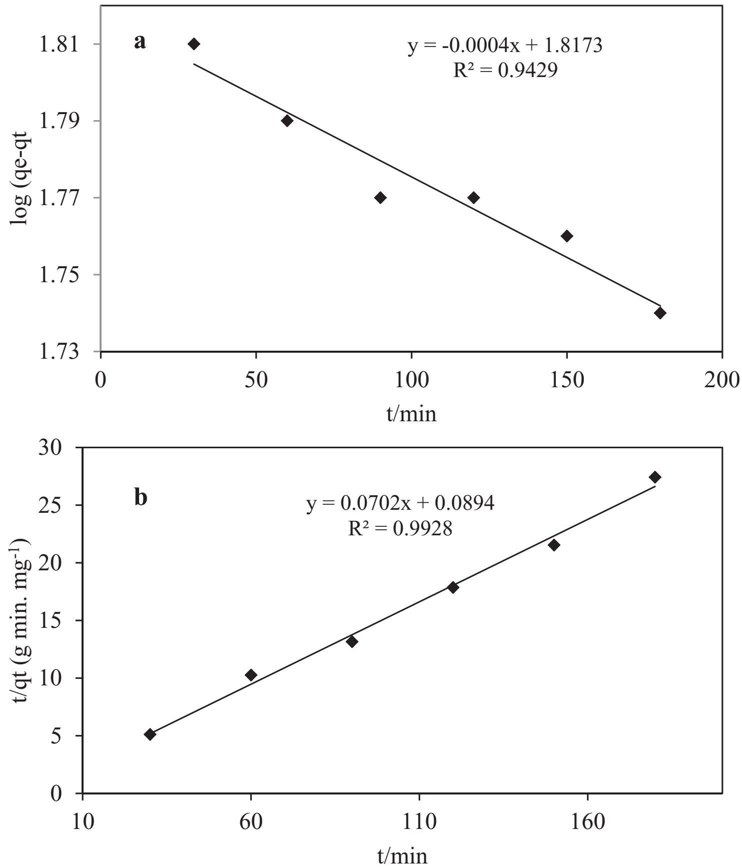


Figure 7. Pseudo-first-order plot (a) and pseudo-second-order plot (b) for cadmium adsorption onto Rooibos shoots.

### *3.3. Adsorption kinetics*

The modeling of the kinetics of cadmium adsorption was studied with Lagergren's pseudo-first-order (Lagergren 1898) and pseudo-second-order (Ho and Mckay 1999) models.

The $q_e$ values for the pseudo-first-order and pseudo-second-order models were obtained from the linear plots of log ($q_e - q_t$) vs. $t$ and $t/q_t$ vs. $t$ (Figure 7(a) and 7(b)). The results from the kinetic models revealed that the data fit well to a pseudo-second-order reaction ($R^2 = 0.9928$) than to a pseudo-first-order reaction ($R^2 = 0.9429$), hence proving that the adsorption of cadmium is governed by chemisorption. Furthermore, the close agreement between the experimental $q_e$ (13.9 mg $g^{-1}$) and calculated $q_e$ (14.24 mg $g^{-1}$) values reflected that the data conformed better to the pseudo-second-order model.

### *3.4. Adsorption equilibrium studies*

The equilibrium data for adsorption of cadmium onto Rooibos shoots was analyzed according to Langmuir (1918) and Freundlich (1906) models.

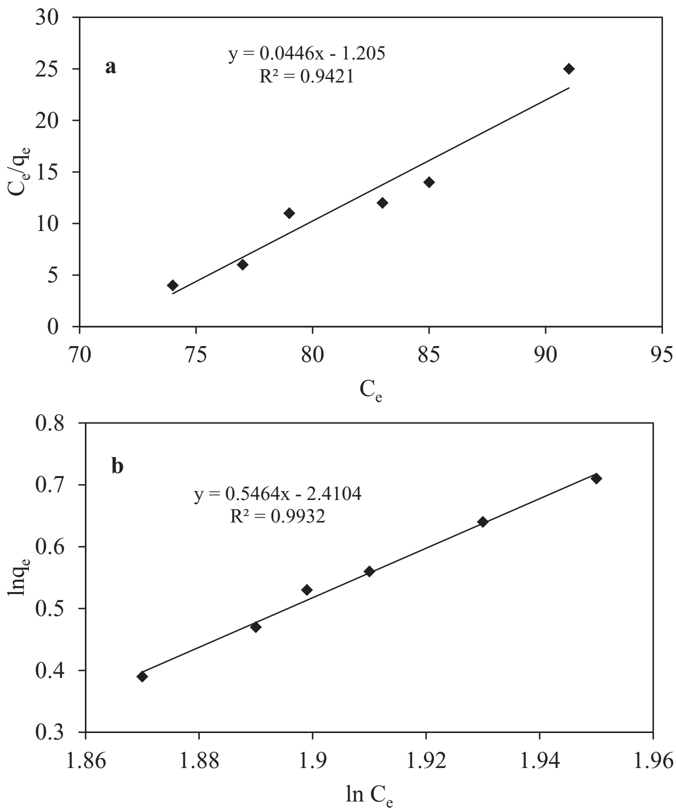


Figure 8. Langmuir isotherm (a) and Freundlich isotherm (b) for cadmium adsorption onto Rooibos shoots.

The Langmuir isotherm plot of $C_e/q_e$ vs. $C_e$ (Figure 8(a)) gave a straight line with an $R^2$ value of 0.9421, suggesting that adsorption conformed to the Langmuir isotherm model. The Langmuir constant, $q_{max}$, was determined to be 22.42 mg g$^{-1}$. The adsorption capacity ($q_{max}$) of Rooibos shoots was comparable or better than many other reported adsorbents (Table 1).

Table 1. Comparison of $q_{max}$ of different adsorbents for cadmium removal.

| Biosorbent | $q_{max}$ (mg g$^{-1}$) | Reference |
|---|---|---|
| Poplar branches | 2.1 | Al-Masri et al. (2010) |
| Grape stalk | 27.88 | Martinez et al. (2006) |
| *Terminalia catappa* Linn leaf | 35.83 | Rao, Anand, and Venkateswarlu (2010b) |
| Pine bark | 7.50 | |
| *Syzygium cumini* leaf | 34.54 | Rao, Anand, and Venkateswarlu (2010b) |
| Tea-industry waste | 11.29 | Çay, Uyanik, and Özaşik (2004) |
| Corncob | 5.09 | Ramos, Jacome, and Rodriguez (2005) |
| Rooibos shoots | 22.42 | This study |

The linear plot of log $q_e$ vs. log $C_e$ (Figure 8(b)) indicated that the adsorption followed the Freundlich isotherm model with an $R^2$ value of 0.9932. The $n$ value was calculated to be 1.83 and falls between 1 and 10, which showed that adsorption of cadmium onto Rooibos shoots powder was favorable. In conclusion, a comparison of the coefficients for isotherm models suggested that the adsorption onto Rooibos shoots was best described by the Freundlich model.

### *3.5. Application on mining effluent*

The performance of Rooibos shoots was evaluated for the removal of cadmium by using effluent from a historical mining dumping site in Mpumalanga, South Africa. Prior to use, samples were allowed to settle for 2 h and were filtered through Whatman No. 1 filter papers. The effluent was found to have a pH of 4.06, total dissolved salts of 98.55 mg $L^{-1}$, and initial cadmium of 24.32 mg $L^{-1}$. An adsorbent dosage of 0.4 g $L^{-1}$ of the effluent and an agitation time of 60 min resulted in 88% removal of the cadmium from the wastewater. The results indicate the efficacy of Rooibos shoots for cleaning environmental wastewaters.

## 4. Conclusion

The adsorption of cadmium onto Rooibos shoots was found to be dependent on factors such as pH, adsorbent dosage, contact time, and concentration of cadmium. The results obtained are:

- pH 5.3 was deduced as optimum for the removal of cadmium.
- Adsorption uptake decreased with increasing biosorbent dosage and increased with increasing cadmium concentration.
- The equilibrium for Rooibos shoots adsorbent was 60 min.
- Adsorption followed the pseudo-second-order model.
- The Freundlich isotherm model best described the adsorption mechanism.

The results from the present study show that Rooibos shoots may be used as a low-cost, eco-friendly, and effective biosorbent to remove cadmium from aqueous solutions.

## Disclosure statement

No potential conflict of interest was reported by the authors.

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