

Experimental investigation of adsorption capacity of anthill in the removal of heavy metals from aqueous solution

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Abstract

In the present work, the adsorption capacity of anthill was investigated as a low-cost adsorbent to remove the heavy metal ions, lead (II) ion (Pb^{2+}), and zinc (II) ion (Zn^{2+}) from an aqueous solution. The equilibrium adsorption isotherms of the heavy metal ions were investigated under batch process. For the study we examined the effect of the solution's pH and the initial cations concentrations on the adsorption process under a fixed contact time and temperature. The anthill sample was characterized using a scanning electron microscope (SEM), X-ray fluorescence (XRF), and Fourier transform infrared (FTIR) techniques. From the SEM analysis, structural change in the adsorbent was a result of heavy metals adsorption. Based on the XRF analysis, the main composition of the anthill sample was silica (SiO_2), alumina (Al_2O_3), and zirconia (ZrO_2). The change in the peaks of the spectra before and after adsorption indicated that there was active participation of surface functional groups during the adsorption process. The experimental data obtained were analyzed using 2- and 3-parameter isotherm models. The isotherm data fitted very well to the 3-parameter Radke–Prausnitz model. It was noted that Pb^{2+} and Zn^{2+} can be effectively removed from aqueous solution using anthill as an adsorbent.

KEYWORDS

adsorption, anthill, characterization, equilibrium isotherm, heavy metal

1 | INTRODUCTION

Indiscriminate disposal of wastewater containing heavy metals has received considerable attention in recent years, primarily due to the fact that their presence in waste stream can be readily adsorbed by aquatic organisms and make them directly enter the human food chain, thus posing a serious health risk to consumers (Lin, MacLean, & Zeng, 2000). Because of the ability of heavy metals to accumulate in living tissues and because they cause damage to these tissues over time, heavy metals are classified as carcinogens. For example, exposure to lead ions can cause anemia, kidney damage, and even untimely death (Mohammed-Ridha, Ahmed, & Raoof, 2017), while zinc ions at elevated concentration result in pancreas damage, osteoporosis, and even death (Wahi, Ngaini, & Jok, 2009). Water or wastewater containing heavy metals requires effective treatment techniques that can completely remove these toxic metals (Yusuff, 2017).

A number of treatment techniques for the removal of heavy metals from waste solution have been reported. These techniques include chemical precipitation, ion exchange, membrane separation, the Fenton-biological method, ultrafiltration, electrochemical degradation, and adsorption. Among these methods, adsorption of adsorbate from fluid onto porous solid material called adsorbent has been

identified as a simple and economical technique (Yusuff, 2017). Adsorbent plays an important role in the adsorption process as it serves as a site for the separation of adsorbate from the fluid. However, the unique feature of an adsorbent is its adsorption capacity, which is usually influenced by the material used and method adopted for its production (Hameed, Krishni, & Sata, 2009). This is because the adsorbent source and its preparation conditions will influence its physical, chemical, and morphological properties. Many adsorbents derived from different sources such as agricultural waste (Yusuff, Olateju, & Ekanem, 2017), naturally occurring materials (Mohamed, Abdelkarrim, Ziat, & Mohamed, 2016), and microorganism (Mohammed-Ridha et al., 2017) just to mention but a few that have been used for the removal of heavy metals from wastewater. In this present work however, anthill, a form of siliceous fireclay, was used as a low-cost adsorbent for the removal of heavy metals from aqueous solution due to its ready availability in Nigeria. According to Akinwekomi, Omotoyinbo, and Folorunso (2012), anthill sample contains metal oxides such as silica (SiO_2), alumina (SiO_2), iron oxide (Fe_2O_3), and the like. However, some of these metal oxides in their pure or composite forms have been used as adsorbents for the removal of contaminants from wastewater (Eletta, Ajayi, Ogunleye, & Akpan, 2016; Fisli, Ridwan, Krisnandi, & Gunlazuardi, 2017). This is the main reason why anthill was chosen as

an adsorbent for the removal of cations from aqueous solution for the present study. Furthermore, to the best of our knowledge, no literature on the adsorption behavior of anthill for the removal of heavy metals from aqueous solution is reported. Thus, in this present study, an anthill sample was thermally activated, characterized, and employed as adsorbent to remove lead (Pb^{2+}) and zinc (Zn^{2+}) ions from aqueous solution. The effects of process parameters affecting the adsorption process, such as medium pH and initial heavy metal concentrations, were investigated. An equilibrium adsorption isotherms study was also carried out and discussed in details.

2 | MATERIALS AND METHODS

2.1 | Materials

The anthill sample used in this study was harvested from a type II anthill situated behind the University's staff quarters, Afe Babalola University, Ado-Ekiti, Nigeria. All chemical reagents and materials used in this study were of analytical grade. About 1,000 milligrams per liter (mg/L) stock solutions of Pb^{2+} and Zn^{2+} were prepared separately by dissolving 3.09 grams (g) of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) and 4.43 g of zinc sulfate ($\text{Zn}(\text{SO}_4)$) in 1 liter (L) of distilled water each. From this prepared stock solution, the desired initial concentrations of each heavy metal ion was prepared for each run, and the concentration was analyzed using atomic absorption spectrophotometer (AAS, Buck Scientific 210VGP, USA).

2.2 | Adsorbent preparation and characterization

The harvested anthill sample was ground by a mechanical grinder into a fine powder. Thereafter, the fine anthill powder was passed through a sieve mesh of 0.5 millimeter to obtain an even finer powder. The anthill powder was then calcined in a muffle furnace at a temperature of 900 degrees Celsius ($^{\circ}\text{C}$) for 2 hours. The adopted heating rate was 10°C per minute, and after the calcination time was attained, the calcined anthill sample was immediately removed from the furnace before its temperature dropped to room temperature. The activated anthill sample was then kept in a sealed glass bottle to prevent contamination with atmospheric moisture. The morphology and topography of the adsorbent before and after adsorption was examined by scanning electron microscope (SEM-JEOL-JSM 7600F). Fourier transform infrared (FTIR) analysis was carried out on both thermally treated and used adsorbents in order to identify various surface functional groups and compared, by using FTIR spectroscope (FTIR-IR Affinity-1S Shimadzu, Japan). Moreover, the chemical compositions of the anthill samples before and after adsorption were determined by X-ray fluorescence machine.

2.3 | Batch equilibrium studies

The batch adsorption process was carried out by bringing into contact the thermally activated anthill adsorbent with an aqueous solution

containing a mixture of Pb^{2+} and Zn^{2+} in a set of conical flasks of 250 milliliters capacity each. The flasks were agitated in a temperature-controlled water bath shaker (SearchTech Instrument) operating at a constant stirring speed of 150 revolutions per minute. The adsorption process was conducted under the following operating conditions: the pH of the aqueous solution was variously 3, 4, 5, 6, 7, 8, and 9, and the initial Pb^{2+} and Zn^{2+} concentrations were 10, 20, 30, 40, 50, and 60 mg/L at a fixed temperature of 35°C for 90 minutes equilibrium contact time. After equilibrium was attained, each sample was filtered to obtain solution containing un-adsorbed Pb^{2+} and Zn^{2+} that was free of the adsorbent, and the concentration of each metal ion was analyzed by atomic absorption spectrophotometer (AAS, Buck Scientific 210VGP, USA). The removal percentage, E_A (%), and the amount of metal ions adsorbed at equilibrium, q_e in milligrams per gram (mg/g), of each metal ion were calculated as follows:

$$E_A = \frac{(C_o - C_e)}{C_o} \times 100\% \quad (1)$$

$$q_e = \frac{(C_o - C_e) V}{W} \quad (2)$$

where C_o and C_e (mg/L) are the initial concentration and concentration at equilibrium, respectively. V (L) is the volume of the solution and W (g) is the mass of activated anthill adsorbent.

3 | RESULTS AND DISCUSSION

3.1 | Adsorbent characterization

The SEM images of the prepared activated anthill before and after adsorption of Pb^{2+} and Zn^{2+} are shown in **Exhibit 1a** and **1b**, respectively. As can be seen in Exhibit 1a, it is obvious that the thermally treated anthill possesses different layers of pores on its surface, which paves the way for heavy metals to be adsorbed. However, some of the pores were blocked due to adsorption of cations on the activated anthill as can be seen in Exhibit 1b.

The chemical composition analysis of prepared activated anthill before and after adsorption as shown in the table in **Exhibit 2** revealed that silica (SiO_2) constitutes the largest percentage in the anthill sample, followed by alumina (Al_2O_3) and zirconia (ZrO_2). However, the percentage of SiO_2 and Al_2O_3 decreased after the adsorption process, as can also be seen in Exhibit 2. This implies that SiO_2 and Al_2O_3 are identified adsorption sites in anthill, and their reduction in total composition after the adsorption process could also be attributed to the fact that the SiO_2 surface contained silanol (OH group), which can interact with Pb^{2+} and Zn^{2+} (Fisli et al., 2017). A similar observation was reported for the adsorption of Pb^{2+} , Cu^{2+} , and Zn^{2+} on soil (Lim & Lee, 2015). This is corroborated by the FTIR analysis. Furthermore, the good adsorption capacity exhibited by the thermally treated anthill sample can also be attributed to the interaction among the metal oxides in the adsorbent as they create several adsorption sites for the adsorbates.

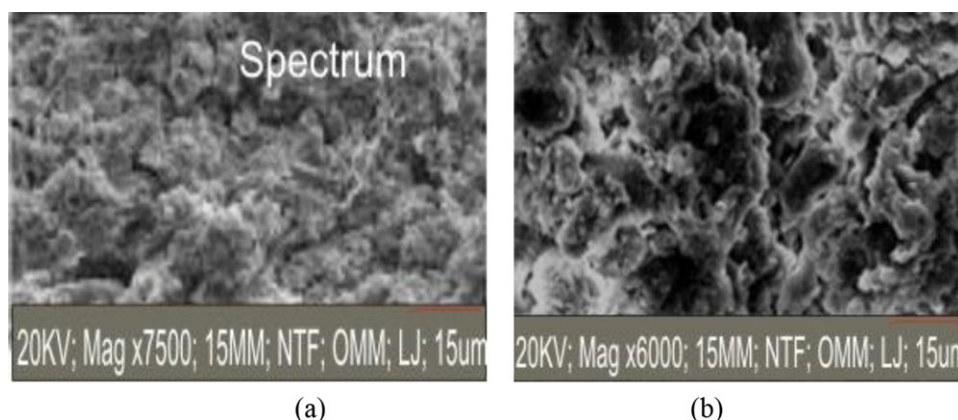


EXHIBIT 1 SEM images of prepared anthill adsorbent (a) before and (b) after adsorption of Pb^{2+} and Zn^{2+} [Color figure can be viewed at wiley-onlinelibrary.com]

EXHIBIT 2 X-ray fluorescence results of prepared activated anthill before and after adsorption process

Chemical composition (wt%)	Before adsorption	After adsorption
SiO_2	58.2	51.0
Al_2O_3	21.6	18.3
Fe_2O_3	2.36	2.66
MgO	4.77	4.72
Na_2O	4.13	4.24
K_2O	0.95	3.91
CaO	0.64	1.10
ZrO_2	6.99	12.4
Other	1.20	3.57

The functional groups present on the surface of the prepared adsorbent before and after adsorption process were identified by FTIR analysis, and their spectra are shown in the table in **Exhibit 3**. Upon the adsorption process, some of the spectra in activated anthill shifted, vanished, and new peaks were formed. The difference in the FTIR spectrum obtained for the prepared activated anthill before and after

adsorption is an indication that there was participation of the surface functional groups during adsorption process (Yusuff, 2017).

3.2 | Effect of process parameters on heavy metals removal

3.2.1 | Effect of pH

The adsorption of Pb^{2+} and Zn^{2+} as a function of the hydrogen ion concentration contained in aqueous solution was examined over a pH range of 3 to 9 as shown in **Exhibit 4**. The removal percentage of both cations decreased with increased pH of the aqueous solution. For both Pb^{2+} and Zn^{2+} , the maximum removal percentage was attained at a pH of 5. The result obtained herein indicates that the adsorption by the anthill of Pb^{2+} and Zn^{2+} would be enhanced at a low pH. Similar observations were reported for Pb^{2+} onto low-cost bio-sorbent (Mohammed-Ridha et al., 2017) and for Zn^{2+} onto soil (Lim & Lee, 2015). The maximum removal percentage of the heavy metals recorded for an acidic medium could be the result of interaction between cations in solution and functional groups on the adsorption sites of anthill (Chiban, Lehtu, Sinan, & Carja, 2009).

EXHIBIT 3 The major absorption band and assignment for anthill adsorbent before and after adsorption

Infrared band	Wavenumber (cm^{-1})		Assignment/Vibration mode
	Before adsorption	After adsorption	
1	3,747.69	–	Si-OH (silanol) vibration mode
2	–	3,693.68	Si-Si-OH or Al-Al-OH stretching vibration
3	1,649.14	1,649.14	H-OH deformation vibration
4	1,085.92	1,105.21	-Si-O stretching
5	–	1,029.99	-Si-O stretching of clay vibration
6	–	914.26	Al-Al-OH deformation
7	785.03	786.96	Al-Mg-OH vibration of clay sheet or O-Si-O deformation vibration
8	688.59	692.44	Coupled Al-O and Si-O out of the plane
9	–	538.14	-Al-O-Si deformation
10	468.70	462.92	Si-O-Al deformation vibration

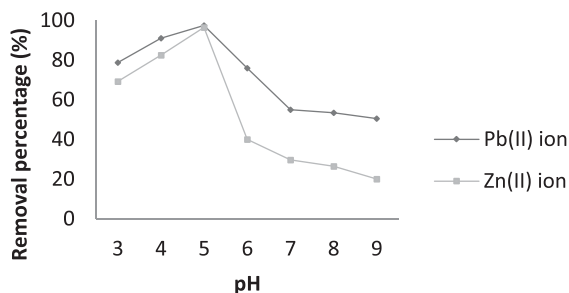


EXHIBIT 4 Effect of pH on removal percentage of Pb^{2+} and Zn^{2+} at fixed initial heavy metal concentration = 50 mg/L, adsorbent dosage = 0.2 g, and temperature = 35°C

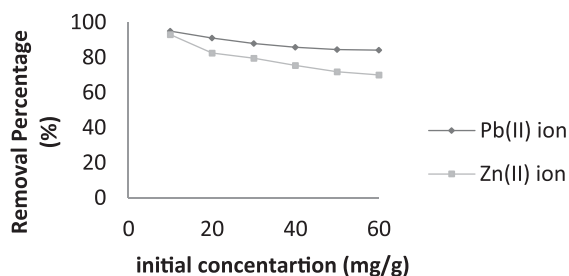


EXHIBIT 5 Effect of initial concentration on removal percentage of Pb^{2+} and Zn^{2+} at fixed pH = 5, adsorbent dosage = 0.2 g, and temperature = 35°C

3.2.2 | Effect of initial concentration of heavy metals

The effects of the initial concentrations of Pb^{2+} and Zn^{2+} on their removal percentages by thermally treated anthill was studied by considering various values of initial concentrations between 10 and 60 mg/L. It was noticed that the removal percentage of cations decreased from 95% to 84.2% and from 93% to 70% for Pb^{2+} and Zn^{2+} , respectively, by increasing the initial concentrations from 10 to 60 mg/L as shown in **Exhibit 5**. This observation revealed that the adsorbent dosage of 0.2 g provided enough active bonding sites for the adhesion of metal ions when the initial concentration was 10 mg/L. However, increasing the initial cations concentrations cause the active bonding sites to become saturated, and the adsorbent capacity becomes exhausted due to non-availability of adsorption sites (Wang & Wang, 2007).

3.2.3 | Adsorption isotherm

In a bid to quantify the amount of adsorbed heavy metals onto activated anthill at equilibrium conditions, two- and three-parameter isotherm models were employed. However, the parameters contained in the selected isotherm models are evaluated by non-linear curve fitting, using Excel Solver and the isotherm model that best describes the experimental results is chosen based on the correlation coefficient (R^2). The experimental data are assumed to be well-predicted by the model the closer the R^2 value comes to unity.

3.2.4 | Two-parameter isotherm model

In this present work, the experimental data for Pb^{2+} and Zn^{2+} adsorption onto activated anthill were fitted to two-parameter isotherm models, the Langmuir and the Freundlich models. The Langmuir and Freundlich isotherm models are given in Equations (3) and (4), respectively, as follows:

$$q_e = \frac{q_{\max} b C_e}{(1 + b C_e)} \quad (3)$$

$$q_e = k_F C_e^{1/n} \quad (4)$$

where q_e (mg/g) is the amount of metal ions adsorbed at equilibrium; C_e (mg/L) is the equilibrium concentration of metal in solution; $q_{\max}$ (mg/g) is the maximum adsorption capacity; b is the Langmuir equilibrium constant; k_F (mg/g (L/mg) $^{1/n}$) indicates the adsorption capacity of the adsorbent; and n is an adsorption intensity.

A dimensionless constant referred to as separation factor (R_L) is applied to ascertain the nature of adsorption by using the Langmuir equilibrium constant (b) and the highest initial concentration of Pb^{2+} and Zn^{2+} (C_0 , mg/L), as given in Equation (5).

$$R_L = \frac{1}{(1 + b C_0)} \quad (5)$$

The separation factor (R_L) can either indicate irreversible adsorption ($R_L = 0$), favorable adsorption ($0 < R_L < 1$), linear adsorption ($R_L = 1$), or unfavorable adsorption ($R_L > 1$).

From the non-curve fitting analysis shown in **Exhibit 6a** and **6b**, the parameters contained in both the Langmuir and the Freundlich models were determined and are presented in the table in **Exhibit 7**. Based on the value of R^2 , Freundlich isotherm provides the best fit to the adsorption equilibrium data of Pb^{2+} , while the adsorption of Zn^{2+} onto activated anthill was best described by the Langmuir isotherm model. A similar observation was reported for adsorption of Pb^{2+} , Cd^{2+} , and Zn^{2+} onto NALCO plant sand (Mohapatra, Khatun, & Anand, 2009). However, the values of separation factor (R_L) obtained for both Pb^{2+} and Zn^{2+} were less than 1 as can be seen in **Exhibit 7**, thus suggesting a favorable adsorption process. Furthermore, by comparing the maximum adsorption capacities of Pb^{2+} and Zn^{2+} on different adsorbents as shown in the table in **Exhibit 8**, activated anthill is found to possess relatively high adsorption capacity, and this implies that it could be regarded as an effective and low-cost adsorbent for the removal of heavy metals from aqueous solution, especially when compared with other forms of clay such as Agbani clay (0.65 mg/g) (Dawodu, Akpomie, & Ejikeme, 2012) and bentonite clay (5.07 mg/g) (Oludotun, 2015).

3.2.5 | Three-parameter isotherm model

For further analyses of the acquired experimental data, three-parameter isotherm models, the Sip and Radke–Prausnitz, were

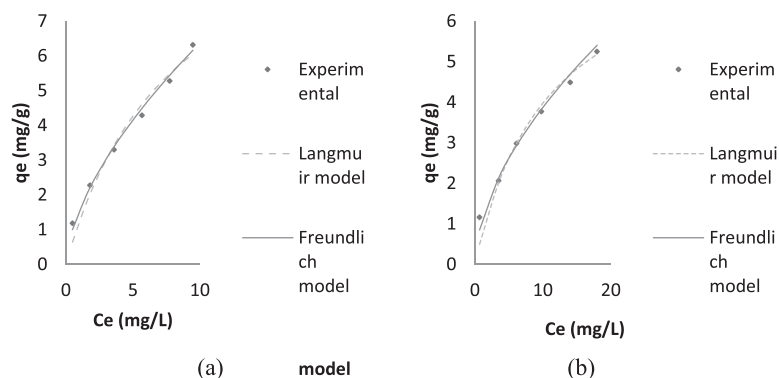


EXHIBIT 6 Two-parameter isotherm models for adsorption of (a) Pb^{2+} and (b) Zn^{2+} onto activated anthill

EXHIBIT 7 Two-parameter isotherm parameters and correlation coefficients for adsorption of Pb^{2+} and Zn^{2+} onto activated anthill

Isotherm	Pb^{2+}	Zn^{2+}
Langmuir		
$q_{\max}$ (mg/g)	11.44	8.39
b (L/mg)	0.119	0.09
R^2	0.9807	0.9842
R_L	0.123	0.156
Freundlich		
k_F (mg/g (L/mg) $^{1/n}$)	1.53	1.04
n	1.62	1.76
R^2	0.9903	0.9783

employed. The Sip and Radke–Prausnitz models are given in Equations (6) and (7), respectively. All of the parameters contained in these two models (evaluated by the non-linear analysis method) and correlation coefficient (R^2) are presented in **Exhibit 9**. However, the plots of q_e against C_e , which display the non-linear regression of the three-parameter isotherm models to the experimental results, and also provide solutions to those models, are depicted in **Exhibit 10a** and **10b**. Based on the value of R^2 , the Radke–Prausnitz isotherm model provides a better fit to the isotherm data than the Sip model.

$$q_e = \frac{q_{\max}^{M_S} (K_S C_e)}{1 + (K_S C_e)} \quad (6)$$

$$q_e = \frac{q_{\max} K_{RP} C_e}{(1 + K_{RP} C_e)^{M_{RP}}} \quad (7)$$

where q_e (mg/g) is the amount of metal ions adsorbed at equilibrium; C_e (mg/L) is the equilibrium concentration of metal in solution; $q_{\max}$ (mg/g) is the maximum adsorption capacity; K_S and K_{RP} are the Sip and the Radke–Prausnitz equilibrium constants, respectively; and M_S and M_{RP} are Sip and Radke–Prausnitz model exponents, respectively.

EXHIBIT 8 Comparison of adsorption capacities of different adsorbents for Pb^{2+} and Zn^{2+}

Adsorbent	Adsorption capacity (mg/g)		Reference
	Pb^{2+}	Zn^{2+}	
Anthill	11.44	8.39	Present work
Red mud	–	14.51	Gupta and Sharma, 2002
Calcareous soil	–	4.587	Mesquita and e Silva, 1996
Agbani clay	0.65	–	Dawodu et al., 2012
Nalco plant sand	21.78	58.28	Mohapatra et al., 2009
Acid soil	–	6.004	Arias, Pérez-Novo, López, and Soto, 2006
Waste beer yeast	2.34	–	Parvathi, Nagendran, and Nareshkumar, 2007
Bentonite clay	5.07	–	Oludotun, 2015

EXHIBIT 9 Three-parameter isotherm parameters and correlation coefficients for adsorption of Pb^{2+} and Zn^{2+} onto activated anthill

Isotherm	Pb^{2+}	Zn^{2+}
Sip		
$q_{\max}$ (mg/g)	222.05	126.44
K_S	0.00035	0.00027
M_S	0.626	0.583
R^2	0.9927	0.9769
Radke–Prausnitz		
$q_{\max}$ (mg/g)	242.71	153.73
K_{RP}	0.051	0.059
M_{RP}	0.381	0.431
R^2	0.9932	0.9782

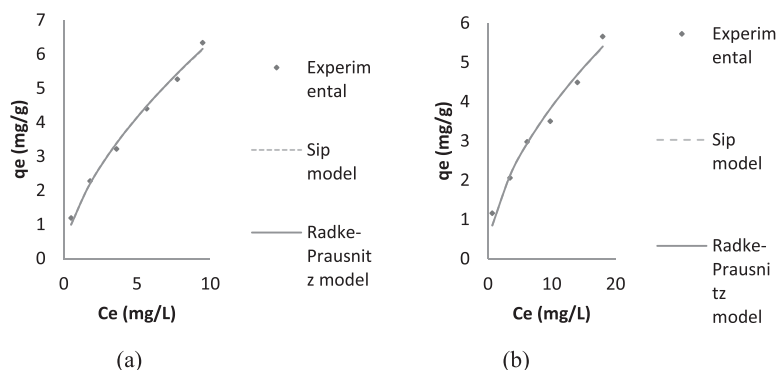


EXHIBIT 10 Three-parameter isotherm models for adsorption of (a) Pb^{2+} and (b) Zn^{2+} onto activated anthill

4 | CONCLUSION

This study revealed that anthill, a naturally occurring material, could be used as a low-cost adsorbent to remove Pb^{2+} and Zn^{2+} from aqueous solution. The adsorption capacity of the cations from aqueous solution descended in order of $\text{Pb}^{2+} > \text{Zn}^{2+}$. The maximum removal percentage of the heavy metal ions was obtained at an optimum pH of 5. The applicability of 2- and 3-parameter isotherm models for the adsorption of heavy metals onto activated anthill was also discussed in detail. The equilibrium adsorption isotherm study revealed that the isotherm data fitted very well to the 3-parameter Radke–Prausnitz model.

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How to cite this article: Yusuff AS, Olateju II. Experimental investigation of adsorption capacity of anthill in the removal of heavy metals from aqueous solution. *Environ Qual Manage.* 2018;27:53–59. <https://doi.org/10.1002/tqem.21536>

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