

Removal of Hg^{2+} , Pb^{2+} , Zn^{2+} and Cu^{2+} ions from Contaminated Water Using *Caladium bicolor* (Wild Cocoyam)

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Abstract

Heavy metal persistence in the environment is a global threat and researchers continue to explore low-cost effective technologies for removing these contaminants from the environment. In this study, the ability of *Caladium bicolor* (biosorbent) to remove lead, zinc, copper and mercury ions from the contaminated water was explored. At 2 ppm metal ion concentration, the biosorbent was able to remove all four metal ions with a low removal efficiency of 79.5% for Hg^{2+} and a high of 99.5% for Pb^{2+} . The effectiveness of the biosorbent, however, decreased when the metal ion concentration was increased beyond 2 ppm. Specifically, the biosorbent's effectiveness was studied at 5, 10, 25, and 50 ppm metal ion concentrations. Interestingly, the decrease in effectiveness with regards to Pb^{2+} was relatively marginal, suggesting a stronger preference of *Caladium bicolor* for lead. In order to determine optimal conditions required for effective adsorption or high metal removal efficiencies, the effect of biosorbent mass and contact time for equilibration of the biosorbent with the metals were also investigated. While 30 min contact time was found to be adequate for effective metal removal, there was no significant difference in the results obtained when 0.5 g vs 2.0 g of the biosorbent was used. FT-IR analyses also revealed that similar functional groups were responsible for the removal of all the four metals studied.

1.0. Introduction

The presence of heavy metals in the environment has become a major threat to plants, animals, and human lives due to their bioaccumulating tendency and toxicity (Wuana and Okieimen 2011). Long-term exposure of these metals result in physical, muscular, neurological diseases including Alzheimer's, Parkinson's and multiple sclerosis (Mohod and Dhote 2013). Thus, the ability to remove these contaminants from the environment is highly essential. A number of conventional methods including chemical precipitation, electrolysis, and reverse osmosis have been employed to remove heavy metals from water and other sources (Arora 2019). However, these methods have disadvantages such as high cost of operation and generation of toxic sludge (Majumder and Gupta 2010). Biofiltration, which involves the use of biomaterials (plants, bacteria, and fungi) has therefore received attention as low-cost but highly effective approach for removing heavy metals from the environment. Since heavy metals are positively charged in aqueous solutions, the presence of compounds that contain either negatively charged functional groups (such as hydroxyl, and carboxyl groups) or groups with lone pair of electrons (such as amines and amides) allow for interaction and adsorption of the metal ions in solution onto the biosorbent. This simple process results in partial or complete removal of the metals.

Aside the simplicity and cost efficiency that biofiltration offers, the technique provides the opportunity to investigate important biological and environmental applications. For example, the interaction of metal ions on functionalized activated charcoal has been exploited in advancing technologies in environmental science and catalysis (Xiao and Thomas 2004). The interaction of metal ions with polycarboxylates has also found widespread application in wastewater treatment for the selective removal of toxic metals

(Bonne, Hofman et al. 2000, Rivas, Pereira et al. 2002). Similarly, the electrostatic interaction of metal ions with carboxylic acid (-COOH) groups is the basis for the construction of assembled layer of hybrid organic/inorganic composite materials (Huang, Tahmasbi Rad et al. 2017, Wang, Wu et al. 2018), which have recently attracted a lot of attention and been used in food packaging (De Vietro, Conte et al. 2017) and cancer therapeutic (Alomari, Almohazey et al. 2019) applications. Biofiltration experiments are therefore relevant and enables an understanding of the chemistry involving the metal ions interactions with various biological materials.

Although several plant materials have been explored as possible biosorbents for removal of heavy metals, the use of *Caladium bicolor* has not been extensively explored despite its ability to remove cadmium (Cd^{2+}) and lead (Pb^{2+}) ions from aqueous solution (Horsfall Jnr and Spiff 2005).

Caladium bicolor is a tuberous perennial plant that grows well in warm, shady areas (Horsfall Jnr and Spiff 2004). A study by Horsfall Jnr and Spiff (2005) shows that the sorption of the metal ions by the *Caladium bicolor* is highly pH and temperature-dependent, and facilitated by the presence of functional groups such as carboxyl, amide, alkyl, carbonyl or hydroxyl ligands on the biomass surface (Horsfall Jnr and Akporhonor 2006). These functional groups have also been shown to be dominant in activated coconut shells that were used to remove arsenic and mercury from water (Emahi, Sakyi et al. 2019), suggesting that *Caladium bicolor* could potentially be used to remove other heavy metals in water beyond cadmium (Cd^{2+}) and lead (Pb^{2+})

In this study, the corm/tuber of *Caladium bicolor* was used as adsorbent to remove Pb^{2+} , Cu^{2+} , Zn^{2+} , and Hg^{2+} from wastewater using water artificially contaminated with the individual metal ions in the laboratory.

2.0. Material And Methods

2.1 Chemicals/ Reagents

Mercury (II) sulphate, lead (II) oxide, zinc granules, copper turnings, were all purchased from BDH Laboratory Supplies, England. Sodium hydroxide was obtained from Fisher Scientific, UK while citric acid and sodium citrate dehydrate were purchased from Daejung Chemicals and Metals Co. Ltd, South Korea.

2.2 Preparation of Contaminated water solutions

100 ppm stock solutions each of Hg^{2+} and Pb^{2+} were prepared from mercury (II) sulphate (HgSO_4) and lead (II) oxide, (PbO) respectively. Zn^{2+} was prepared from zinc granules and Cu^{2+} was prepared from copper turnings into a 500 ml volumetric flask. The concentrations of the stock solutions were quantified using Inductively Coupled Plasma-Mass Spectrometer (ICP-MS Nexion 2000, Perkin Elmer). Working solutions were later prepared from the stock solutions via serial dilutions for subsequent experiments.

2.3. Preparation of *Caladium bicolor* biomass.

Fresh *Caladium bicolor* corm/tubers were harvested from the campus of University of Energy and Natural Resources in Sunyani, Ghana. The tubers were thoroughly washed with tap water followed by distilled water and oven-dried at 60°C for 24 hours. The dried samples were grinded, and the pH of an aqueous solution of the sample determined using pH meter.

2.4 Activation of *Caladium bicolor* biomass.

The grinded *Caladium bicolor* sample was soaked and constantly stirred in 0.3 M sodium hydroxide (NaOH) solution for 2 hours. The NaOH-biomass sample was then thoroughly rinsed with citric acid buffer of pH 6.01 in order to restore and maintain the pH of the *Caladium bicolor* prior to the activation process. The biomass was oven-dried at 60°C for 24 hours to complete the activation.

2.5 Biofiltration experiments

Ability of the activated *caladium bicolor* biosorbent to remove the above metals from the artificially contaminated metal solutions were studied under three different conditions described below;

2.5.1 Concentration Variation

Metal ion solutions of 2 ppm, 5 ppm, 10 ppm, 25 ppm and 50 ppm were prepared from respective stock solutions via serial dilution. 50 ml of each working solution was mixed with approximately 1.00 g of the activated *Caladium bicolor* and equilibrated at RT, with gentle stirring, for 30 min. The mixture was then filtered, and the concentration of each metal remaining in the filtrate was quantified using Inductively Coupled Plasma- Mass Spectrometer (ICP-MS Nexion 2000, Perkin Elmer).

2.5.2 Mass Variation

Approximately 0.50 g and 2.00 g each of the activated *Caladium bicolor* was mixed with 50 ml of 2 ppm working solutions of each contaminated metal solution. The mixture was equilibrated at RT, with gentle stirring, for 30 min, and the filtrate quantified as described in 2.5.1.

2.5.3 Contact time Analysis

The procedure described in 2.5.1 was repeated using 2 ppm working solutions of each contaminated metal solution, but with varying incubation times - 5 min, 15 min, 30 min, 1 h, 2 h, and 5 h.

2.6 Estimation of metal ion removal efficiency

The amount of each metal ion adsorbed or removed by the *Caladium bicolor* (biosorbent) in the three different experiments were estimated using equation 1.

$$\% \text{Removal} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

Where C_i and C_f indicates the concentration of the metal ion solutions before and after adsorption respectively.

2.7 Functional group determination

The functional groups present on the grinded *Caladium bicolor* biosorbent sample before activation (Raw Sample), after activation with NaOH, and after biofiltration were characterized using Fourier-Transform Infra-Red (FT-IR) spectroscopy.

3.0. Results

3.1 Removal Efficiency

3.1.1 Concentration Variations

Figure 1 shows a graph of the efficiency of the *Caladium bicolor* biofilter towards the removal of metal ions at different concentrations of the respective contaminated metal ion solutions.

As can be observed from the graph, highest percentage removals were recorded in the solutions containing 2 ppm of the metal ions, followed by 5, 10, 25, and 50 ppm respectively. This is expected because a fixed mass of the biomass (1.0 g) was used in each experiment. Since this amount of biomass will have approximately the same number of adsorption or binding sites, it makes sense that less sites may be available for adsorption as the metal ion concentration is increased. With regards to the specific metals, the removal of lead (Pb^{2+}) was relatively and consistently very high, with highest removal percentage of 99.5% at 2.0 ppm initial Pb^{2+} solution, and lowest of 85.9% at 50.0 ppm initial Pb^{2+} solution. These results also confirm the fact that lead typically adsorbs more strongly to organic materials compared to other heavy metals (Usman 2008).

3.1.2 Effect of biomass quantity (mass) on metal adsorption

It may appear obvious that increasing the quantity (mass) of the *Caladium bicolor* will increase the amount of metal ions adsorbed. Results of this study is presented in Fig. 2.

Apparently, there is no direct correlation between the mass of the biofilter and the amount of metal ion adsorbed. Whereas a lower mass (0.5 g) of the biofilter removed a relatively higher concentration of Cu^{2+} ions (94.7%) than a 2.0 g sample (percent removal = 91.7%), the reverse trend and rather marginal differences in percent removal in the case of the other three metals (Pb^{2+} , Hg^{2+} , and Zn^{2+}) does not warrant a clear conclusion to be drawn. The adsorption process may be more dependent on the kinetics and chemical behavior of the different metals at biofilter-metal interface and not just the adsorption capacity of the biofilter. Follow up experiments will involve extensive characterization of this metal-biofilter interface and the results will be published elsewhere.

3.1.3 Effect of contact time on adsorption

Equilibration or the extent of contact of the metal ions with the biofilter was studied at different time points, and Fig. 3 presents the effect of this contact time on the removal efficiency of the biofilter towards the various metals.

With the exception of Cu^{2+} , the extent of contact of the metals with the biofilter did not significantly affect the adsorption or removal efficiency. This suggests that the 30 min incubation time used in the biofiltration processes was adequate to ensure maximum removal of the metal ions. The slight decrease in adsorption of Cu^{2+} with longer incubation time may be indicative of a different mode of interaction of the Cu^{2+} to the biofilter.

3.2 FT-IR Spectroscopy

3.2.1 Determination of functional groups on *Caladium bicolor* biofilter

The functional groups present on the *Caladium bicolor* biofilter were characterized via FT-IR spectroscopy. Since the biofilter was alkaline-activated prior to use in the biofiltration experiments, it was important to investigate any possible structural changes on the biofilter, which may have affected the metal adsorption. Interestingly, the IR spectrum of both the raw sample (unactivated) and the activated sample were similar (Fig. 4). This may not be surprising, however, since the pH of the solution of the biofilter was restored to its initial (before activation) pH prior to the biofiltration experiments.

Caladium bicolor is reported to chiefly contain hydroxyl, carboxyl, amide, alkyl and carbonyl groups (Horsfall Jnr and Akporhonor 2006), which is consistent with the results obtained here. The broad and strong band observed at 3500-3200 cm^{-1} in the FT-IR spectrum of the raw *Caladium bicolor* sample as shown in Fig. 4 is due to the O-H stretching vibrations possibly from phenol, alcohols or carboxylic acid groups. This same band was also observed for the activated *Caladium bicolor* sample (Fig. 4) with a slight decrease in intensity. A slightly strong and sharp band was also observed around 1600 cm^{-1} for both the raw and activated *Caladium bicolor* samples, which is due to the C=C stretching vibration of either an alkene or an aromatic ring. A strong and sharp band was also seen around 1000 cm^{-1} for both the raw and activated *Caladium bicolor* samples although the spectrum of the raw sample had a slightly greater intensity compared to the activated sample. This peak is due to the C-O stretching vibration of either alcohols, phenols, ether, ester, or carboxylic acid. There was no alkene =C-H stretching vibration observed around 3100 cm^{-1} even though a C=C stretching was observed; this is due to the fact that all the C=C components of the *Caladium bicolor* that were captured during the FT-IR Spectroscopy analysis were tetra substituted.

3.2.2 FT-IR spectra of *Caladium bicolor* biofilter after adsorption of metal ions

Figure 5 shows the FT-IR spectra of the activated *Caladium bicolor* sample after the biofiltration of Hg^{2+} , Pb^{2+} , Zn^{2+} and Cu^{2+} metal ions. The O-H stretching vibration around 3500-3200 cm^{-1} was observed to be more intense after the filtration of Hg^{2+} and Cu^{2+} metal ions and reduced slightly after the adsorption of Pb^{2+} , and Zn^{2+} metal ions.

The C=C band which was observed around 1623 cm⁻¹ for the activated *Caladium bicolor* sample increased slightly in wavenumber to 1629 cm⁻¹, 1637 cm⁻¹, and 1630 cm⁻¹ after the adsorption of Cu²⁺, Pb²⁺, and Zn²⁺ respectively, but shifted slightly to 1616 cm⁻¹ after the adsorption of Hg²⁺. The band around 1000 cm⁻¹ which is due to the C-O stretching vibration as mentioned earlier experienced a slight decrease in intensity after the biofiltration of the various metal ions (Fig. 5) compared to the activated *Caladium bicolor* sample (Fig. 4).

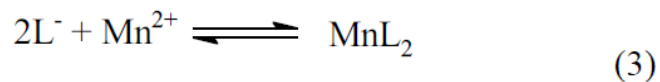
4.0. Discussion

Caladium bicolor contains mainly organic residues, which are made up of several polar functional groups (Ligands) that are responsible for chemical bonding as well as cationic exchange capacities of the biomass (Horsfall Jnr and Spiff 2004). This is consistent with the spectra obtained in our FT-IR characterization of the biofilter (Fig. 4). Activating the *Caladium bicolor* biomass exposes the binding sites of these ligands for better chemical bonding and ion exchange effects as described in equation 2.



Where L represents ligand.

Hence the general equation for the sorption of metal ions by the *Caladium bicolor* can be represented by equation 3 (Horsfall Jnr and Spiff 2004);



Where L and Mn²⁺ represents Ligand and metal ions respectively.

The likely mode of interaction of the metal ions of interest with the *Caladium bicolor* biomass based on the given equations could be via complexation or ion exchange or both as stated in literature. The effectiveness of *Caladium bicolor* to remove the metals investigated in this study can be ranked as Pb²⁺ > Cu²⁺ > Zn²⁺ > Hg²⁺ (Fig. 1). This observation implies that the adsorptive removal of the metals were not necessary based on the ionic size of the metals. This observation is also consistent with some literature reports. In studying the interaction of carboxylic acid (-COOH) groups with various divalent metal ions, Bala, Prasad et al. (2007) observed that Pb²⁺ consistently exhibited stronger binding than Cd²⁺ despite the smaller ionic radius of the latter. Thus the strength of interaction and therefore removal efficiencies of the metals may be influenced by the type of geometry formed between the organic compounds in the *Caladium bicolor*, containing the key functional groups, with the metal ions.

Generally, it is also observed that increasing the concentration of the metal ion solutions resulted in decreased adsorption of the various metal ions (Fig. 1). This can be attributed to the fact that as the concentration of the metal ions increases, the surface area of the *Caladium bicolor* becomes saturated with the metal ions (Adebowale, Unuabonah et al. 2006, Wang, Guangyan et al. 2015), and thus making it difficult for the *Caladium bicolor* to take up more of the metal ions. An exception was observed in the case of Hg^{2+} , which showed relatively higher removal efficiency (20.8%) at 50 ppm compared to 18.4% at 25 ppm. This could be due to slightly different adsorption profile for Hg^{2+} , which will be further investigated. Throughout the concentration variation experiments, 1.0 g of the *Caladium bicolor* was used and this generally resulted in very high adsorption of the metals as estimated from the percentage removal efficiencies especially for the 2 ppm. It is arguable if 1.0 g of the biomass was optimal to provide ample adsorption sites to uptake the metals from solution. To address this query we decided to repeat the experiment using much higher (2.0 g) and less (0.5 g) biosorbent. Interestingly, the performance of the two different biosorbent masses was not statistically different. As depicted in Fig. 2, both masses showed significantly high and comparable removal efficiencies in the biofiltration of all the four metals studied. Since both masses recorded no significant differences, we decided to investigate the effect of contact time on the removal efficiencies of the various metals using 1.0 g of the biomass. As shown in Fig. 3, the adsorption characteristics of Pb^{2+} , Hg^{2+} , and Zn^{2+} were quite similar. In all cases, highest removal efficiency was achieved within the first 30 min and remained relatively unchanged after 5 h. In the case of Cu^{2+} , however, highest removal was recorded within the first 5 min but this fluctuated between 84% and 64% during the 5 h time of contact. This could likely be due to unexpected temperature elevations (Conway, Angerstein-Kozłowska et al. 1978) and certain physisorption behavior in which some of the metal ions initially adsorbed get desorbed over time. This therefore implies that time is a key factor to consider when using *Caladium bicolor* for water treatment.

The functional groups observed from the FT-IR characterization analysis was virtually the same as reported in literature (Horsfall Jnr and Akporhonor 2006). The shifting of wavenumber and band intensities after biofiltration of the C-O, O-H, and C=C functional groups from the IR spectra confirmation that these functional groups were indeed responsible for the chelation, ion exchange and complexation for a successful removal of the metal ions.

Conclusion

We have successfully utilized the corm of *Caladium bicolor* to remove various heavy metal ions (Hg^{2+} , Pb^{2+} , Zn^{2+} and Cu^{2+}) via simple biofiltration. Although the biosorbent was effective at removing all four metal ions studied, it showed greater affinity for lead (Pb^{2+}). The order of preference of the biosorbent towards the metal ions were in the order $Pb^{2+} > Cu^{2+} > Zn^{2+} > Hg^{2+}$. From an initial 2 ppm lead polluted water, the biosorbent was able to remove 99.5% within 30 min of equilibration. As may be expected, allowing more time for equilibration of the biosorbent with Pb^{2+} led to increase in percent removal although the increment was very marginal. At 60 min, the percent removal remained 99.4% and only increased to 99.6% after 2 h of equilibration. We attribute this to the fact that the biosorbent may have already been saturated at 30 min and therefore there was not much adsorption sites left for further

adsorption. Interestingly, the different metal ions showed slightly different patterns in removal efficiencies with regards to increasing the metal ion concentration, changing mass of the biosorbent, and varying biosorbent-metal ion equilibration times. This observation suggests that although the same compound or functional group was present on the biosorbent to interact with the metal ions, the kinetics and chemical behavior of the metal ions, including their ionic sizes and the type of geometry formed with the organic compounds may have played key roles in the removal process. Additional work will, however, be needed to provide details of the chemistry involved in these interactions. The fact that *Caladium bicolor* is non-edible, readily available, and its high metal removal efficiency as recorded in this study makes it sustainable, cost effective material for use in designing wastewater treatment technologies, especially towards the removal of lead.

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Figures

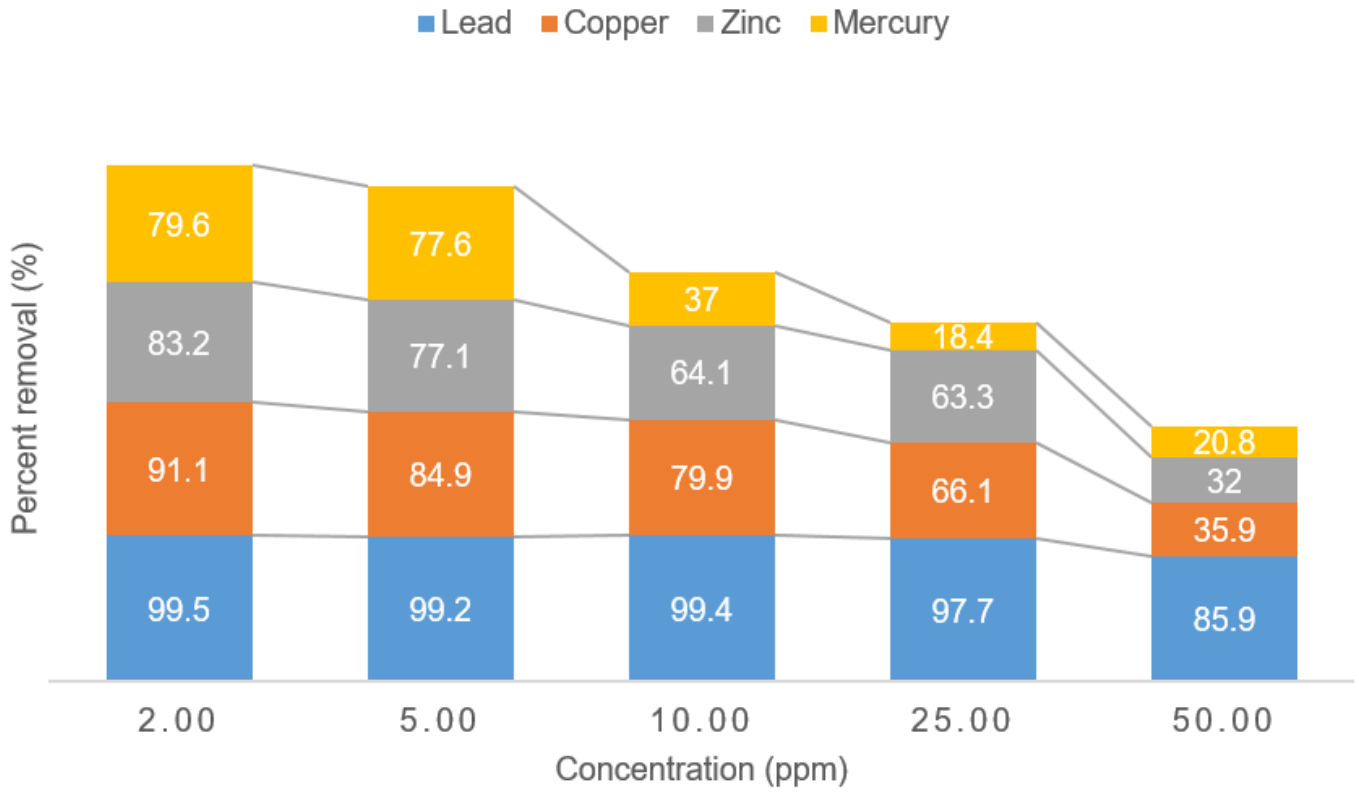


Figure 1

A graph of the efficiency of Caladium bicolor in removing different concentrations of metal ions in aqueous solution. Values in the shaded regions indicate the respective percentages of the metal ions removed from the initial contaminated solutions. From bottom to top are bars representing lead (Pb²⁺), copper (Cu²⁺), zinc (Zn²⁺), and mercury (Hg²⁺) respectively.

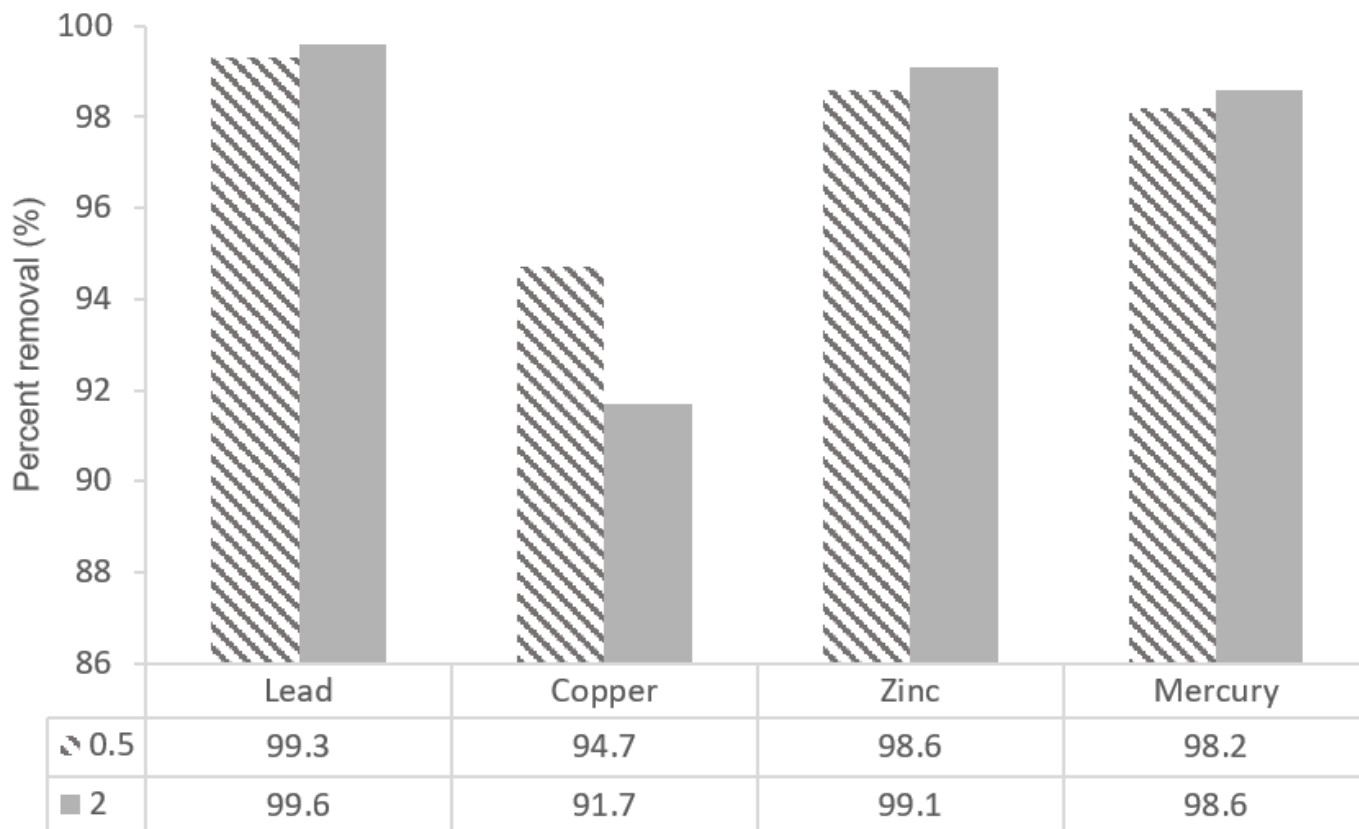


Figure 2

Effect of mass on the removal efficiency of Pb^{2+} , Hg^{2+} , Cu^{2+} , and Zn^{2+} . Two different masses (0.50 g and 2.0 g) of the *Caladium bicolor* biofilter were used in this study. A fixed metal concentration of 2 ppm was used in each case.

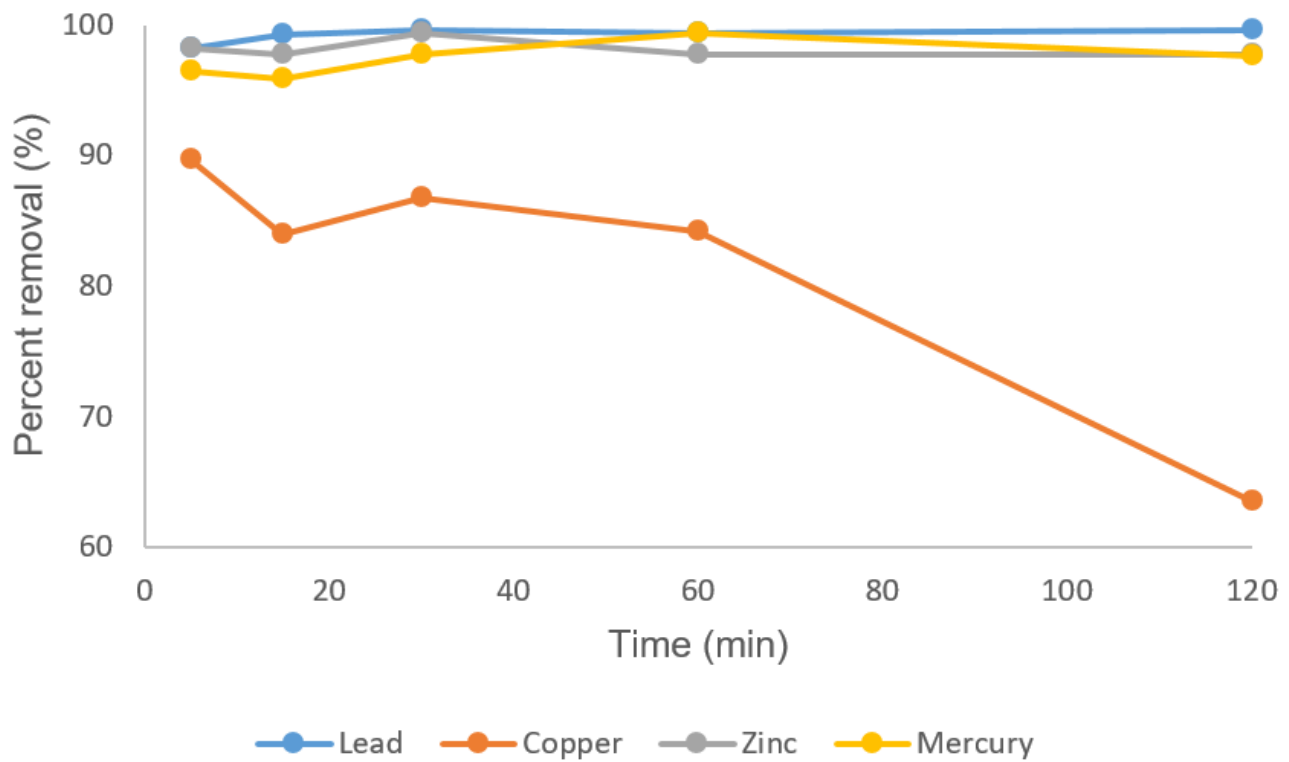


Figure 3

Effect of contact time on adsorption. 1.0 g of the *Caladium bicolor* biomass was equilibrated with 2 ppm of each of the metal ions for 5 min, 15 min, 30 min, 1 h and 2 h.

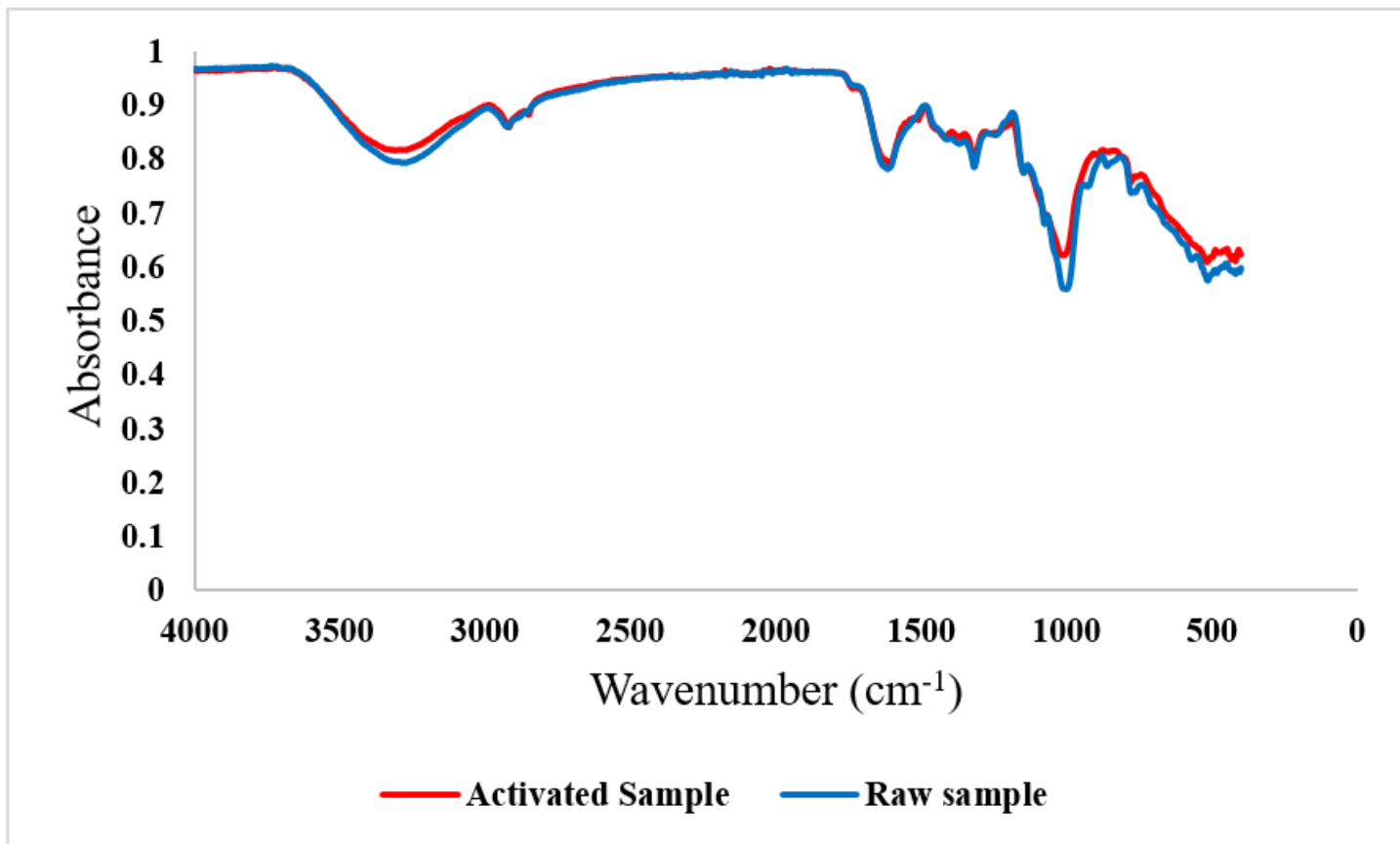


Figure 4

FT-IR Spectra of raw versus activated Caladium bicolor.

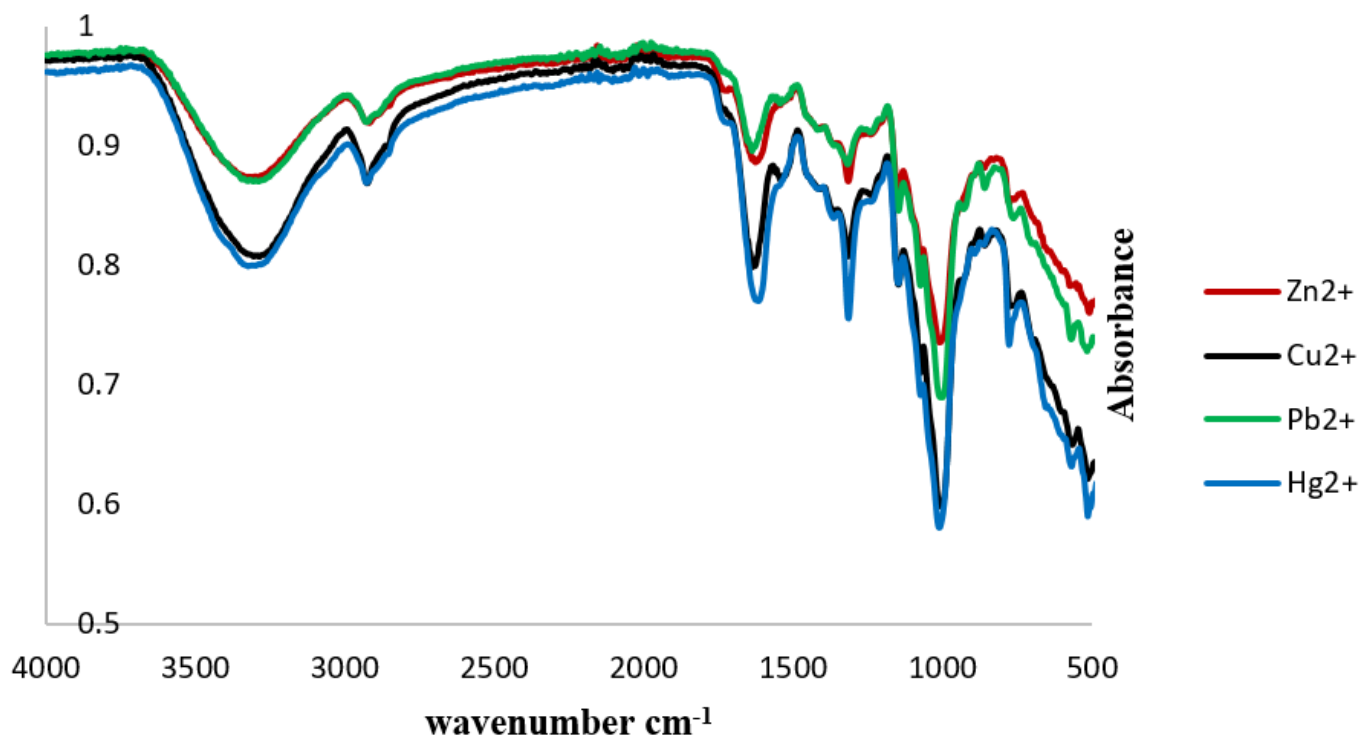


Figure 5

FT-IR spectra of the Caladium bicolor after the biofiltration experiments. Each spectra is based on the the FT-IR characterization of the activated Caladium bicolor powder after it has been used to filter the respective metal ion solution. Each sample was thoroughly dried at RT to remove excess moisture before taking the spectrum.