Research Article

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# Compost material for high-rate Cu²⁺ and Cd²⁺ removal from polluted water: batch experimental study

## Abstract

The present study investigated the adsorption of Cu, Cd from polluted water onto different compost materials. The treatment has been attempted via batch experiments under various solution concentrations. It probes mainly three adsorbents. The metal initial concentration solutions were ranging from 0 to 50000μg/g. Characterizations of the adsorbents was one of the key focal areas of experiment investigation. Adsorbents were characterized by using Fourier transform infrared spectroscopy revealed that the metal binding is perhaps mainly due to the ionic interaction of the metal cations with the carboxyl groups in the humic substances. Scanning electron microscope was utilized for morphological analysis of the adsorbent and revealed changes in compost composition that could be related to the mechanism of metal adsorption. Energy dispersive X-ray spectroscopy used before and after the metal was loaded, thus gave evident on compost adsorption of heavy metal and metals involvement in the interaction with metal ions by ion exchange. The initial and after adsorption concentrations of the metals were determined using Atomic Absorption Spectroscopy. Higher amounts were adsorbed from Cu followed by Cd. The data fitted well to Langmuir model.

**Keywords:** heavy metal, compost, adsorption, langmuir

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## Introduction

Water pollution is the contamination of water bodies such as lakes, rivers, oceans, and groundwater caused by human activities, which can be harmful to organisms and plants which live in these water bodies. Water pollution by toxic heavy metals through the discharge of industrial waste is a worldwide environmental problem. The presence of heavy metals in streams, lakes, and groundwater reservoirs has been responsible for several health problems with plants, animals, and human beings.[1] Copper is widely applied in the electrical wiring, selenium rectifier, plumbing, gear wheel and roofing industries for its electrical and thermal conductivity, good corrosion resistance and ease of installation and fabrication. The potential sources of copper in industrial effluent are from pulp and paper board mills, plating baths and metal cleaning, the fertilizer industry and wood pulp production.[2] Cadmium is one such heavy metal which is added to water and soil through the effluent from battery, catalyst, paint, mining and many other industries. It affects human being adversely when injected into human bodies. It can cause both long term and short-term diseases in human beings and other aquatic life.[3] In recent decades, the annual global release of heavy metal reached 22,000 tons (metric tons) for cadmium, 939,000 tons for copper, 783,000 tons for lead and 1,350,000 tons for zinc.[4] Consequently, it became very important to remove such contaminants from industrial wastewater before they are discharged into the environment. In recent times filters have been developed to remove heavy metals from wastewater, and the spending on filtration is estimated to increase from $17 billion to $75 billion by 2020.[5] The removals of these hazardous materials may be performed using various techniques, including precipitation, membrane filtration, ion exchange, sorptive flotation and adsorption. However, these methods are known for their high cost and ineffective when dealing with low heavy metal concentration. While a number of researchers have attempted to resolve this problem through the use of different technologies, an efficient and cost-effective approach for heavy metal removal remains a challenge. Among many methods, adsorption techniques have been found appropriate for wastewater treatment because of their cost effectiveness and uncomplicatedness. Adsorption has been recognized as an effective process in most of the industrial water and wastewater treatment.[6] Adsorption is the process of the attraction and accumulation of gas or liquid particles on the surface of another substance. Whereas the absorbed particles are called the adsorbate, the substance applied for adsorption is call the adsorbent. The foremost benefits of biosorption technology are its efficacy in reducing the concentration of heavy metal ions to very low levels and the use of low-cost biosorbent materials. In an effort to develop a useful, easy to use, eco-friendly and low-cost application, the study conducted attempted the removal of heavy metal by compost material. Compost has several benefits, such as improving soil fertility, soil structure, increasing plant fertility, increasing water absorption capacity. Besides its use as fertilizer, compost also can be applied as a heavy metal adsorbent. It would thus be of interest to study whether these materials will be feasible for metal removal. This paper evaluates the adsorption of Cd²⁺, Cu²⁺ and by using compost material as an adsorbent application. Batch experiments were carried out to examine the ability of different compost material of removing heavy metal from industrial waters.

## Materials and methods

### Preparation of adsorbent

Adsorbents were provided by laboratory of bio combustibles from SzentIstvan University, Gödöllő (Hungary). Each compost was ground and sieved into fractions below 2mm. The fractions collected

below mesh size of 2mm were designated for the characterization and biosorption investigations without any pre-treatment. Felgyő compost is green waste and sewage sludge. Garé is a communal sewage sludge, slurry mud and chicken manure with straw. Sioagárd is green biomass/ bio waste. The chemical composition evaluated by SEM and depicted in (Tables 1 & 2). The particular weight of compost sample was one gram. 13 samples were prepared for single set, and 3 replications were conducted to carry the test for one single metal followed by the same steps for other composts besides the heavy metals. Finally, every set of samples has 10ml of concentration solution per each bottle plus one gram of compost. The concentration solution range from 0 to 50000μg/g of one single metal and the homogeneous mixture was ready to be investigated.

## Statistical analysis

The result was presented as mean standard deviation.

## Quality control

The distilled water was digested and analyzed with every sample group to track any possible contamination source and obtain a baseline value. Triplicate runs for batch mode adsorption experiments were made for each adsorbent to determine the relative deviation of the experiments. The adsorption of metal ions on the adsorbent material remained almost constant with the relative deviation of the order of **±2%.**

## Adsorption experiments

After each adsorption, the residual Cd and Cu were determined by digesting the filtered synthetic wastewater followed by AAS analysis as a standard method. The percentage of compost was estimated using the following equation:

$$R\ (\%) = (C_o - C_e / C_o) \times 100 \quad (1)$$

Where $C_o$ and $C_e$ are metal ions concentrations (mg/L) before and after adsorption respectively. M is the weight of the adsorbent in grams.

## Effect of agitation speed

To determine the effect of agitation speed, 10mg of compost was added to 13 conical flasks containing 10mL of pre-pared sample with known metal concentrations varied from 50mg/L to 50000μg/g. The agitation speed was set at 6000 rpm for 7 minutes in centrifuge for each flask at 25°C. Thereafter, an aliquot was taken for AAS analysis.

## Preparation of synthetic wastewater

Cd and Cu added separately to distilled water in the laboratory and the value of concentration was determined in a range from 0 to 50000μg/g. The stock solution of 1000 mg L$^{-1}$ Cu²⁺ was prepared by dissolving ($CuSO_4 5H_2O$) and Cd ($NO_3)_{2·4}H_2O$ in different flasks of 1.0 L deionized water and further diluted to the desired concentration with deionized water mixture was adjusted to the desired value then the concentration solution filled 13 bottles to make an initial concentration of (0–50000μg/g) for each metal. The chemicals used throughout this study were of analytical reagent grade provided by Reanal Ltd, Hungary. The individual metal ion concentrations in solution were analyzed using an atomic absorption spectrophotometer (AAS) Varian Spectra AA-20 at the Analysis Service Unit of Szentistvan University, Hungary.

## Experimental set up

Batch adsorption experiments were carried out by shaking a series of samples containing same amounts of different concentration solution with mixture of 3 different composts used to sorb the heavy metal separately. The compost materials used were mixed with concentration range of (0, 50, 100, 250, 500, 100, 250, 500, 10000, 25000, 30000, 40000, and 50000μg/g) of Cu and Cd. After the composts has been collected, the product was ground and sieved to 2mm whichtransferred to small flask with screw cap which contained 1g compost plus 10 ml of concentration solution, and then immerged in a shaking machine as a complete set containing 13 samples at a constant speed of 125rpm. Shaking every set of experiments for 24hours. All the experiments were performed in triplicate method and the adsorbate and adsorbent were separated by high speed centrifugation at 6000 rpm for 7 minutes. In this stage of the experiment the adsorbent particles were separated from the suspensions by filtration through 0.4μm filter paper. The residual concentration of heavy metals was determined by the Atomic absorption photometer. The measured data were fitted to Langmuir function.

# Result and discussion

## Analysis and characterization of compost

**Scanning electron microscopy (SEM):** The surface morphology of the three composts were examined by SEM before and after the heavy metals were loaded. For the magnification, the scanning electron microscope was set at x35 and x50 magnification, while the accelerated voltage was set at 5kV in order to obtain high-resolution micrographs at different adsorbent spots. Figures 1A-1CFelgyő Garé, Sioagárd compost respectively) shows how an irregular surface, which is heterogeneously shaped and cracked probably favours the biosorption of heavy metals on different parts of the biosorbents; making it a propitious adsorption technique.From (Tables 1 & 2)it was observed that after adsorption of heavy metals, ion percentage of other elements such as Na, K, and Ca was decreased. Amount of exchangeable cations Na+ was reduced after metal adsorption.[7]and 1.052 % $Cu^{2+}$ and Cd 1.36 appeared. In this case other elements may be involved in the ion exchange process with the Cu²⁺and Cd ions. From (Tables 1 &2) the analysis of adsorbents also indicated that carbon was a major component in compost, followed by oxygen and nitrogen. Because of the heterogeneous structure which also led to differences in the elemental composition when different parts of the sample surface were measured the composition of the compostsin the chemical elemental analysis was predicted to illustrate that the chemical constituents could stimulate the heavy metal adsorptive capacity of the adsorbents Figures 1A-1C & 2A-2Crevealed the morphological changes in raw composts, due to treatment of Cu²⁺ and Cd.

**Energy dispersive X-ray spectroscopy (EDX):** EDX analysis provided direct confirmation for the copper and cadmium sorption onto the compost materials. The samples were coated with gold prior to analysis to avoid electron charging. Resulting interaction with metal ions, the features of Cu²⁺ and Cd can be observed in each EDX spectrum of the biosorbents. The EDX spectrum before and after exposure with Cu²⁺and Cd is shown in Figures 3 & 4 respectively. The disappearance of some metal features after metal ions adsorption may be due to ion-exchange mechanism, this is also incurred with.[8]The appearance of and Cd Cu²⁺ features can be observed on EDX spectrum at different energy values indicates that the biosorbents studied were able to bind metal ions.

**Table 1** The chemical composition of compost felgyő before and after loaded with Cu

| Sample magnification | Row compost x35 | | Row compost x50 | | Cu loaded x35 | | Cu loaded x50 | |
|---|---|---|---|---|---|---|---|---|
| Element | At. ratio | Wt % | At. ratio | Wt % | At. ratio | Wt % | At. ratio | Wt % |
| C | 41.22 | 32.164 | 44.708 | 35.233 | 27.487 | 19.867 | 29.235 | 21.398 |
| N | 12.9 | 11.738 | 10.048 | 9.235 | 14.03 | 11.83 | 13.81 | 11.79 |
| O | 38.163 | 39.667 | 38.043 | 39.936 | 45.016 | 43.342 | 44.618 | 43.502 |
| Na | 0.267 | 0.399 | 0.197 | 0.297 | — | — | — | — |
| Mg | 0.395 | 0.623 | 0.357 | 0.57 | 0.563 | 0.823 | 0.535 | 0.792 |
| Al | 0.771 | 1.351 | 0.806 | 1.428 | 2.51 | 4.08 | 2.26 | 3.72 |
| Si | 2.759 | 5.035 | 2.552 | 4.703 | 8.016 | 13.55 | 7.158 | 12.25 |
| P | 0.444 | 0.893 | 0.442 | 0.899 | 0.168 | 0.313 | 0.18 | 0.339 |
| S | 0.288 | 0.599 | 0.352 | 0.74 | 0.055 | 0.107 | 0.096 | 0.188 |
| Cl | 0.124 | 0.286 | 0.097 | 0.225 | — | — | — | — |
| K | 0.69 | 1.753 | 0.639 | 1.64 | 0.391 | 0.92 | 0.329 | 0.783 |
| Ca | 1.652 | 4.301 | 1.302 | 3.423 | 0.92 | 2.21 | 0.98 | 2.38 |
| Fe | 0.328 | 1.19 | 0.456 | 1.672 | 0.57 | 1.91 | 0.53 | 1.81 |
| Cu | — | — | — | — | 0.275 | 1.052 | 0.27 | 1.046 |

**Table 2** The chemical composition of compost felgyő before and after loaded with Cd

| Samplemagnification | Raw felgyőcompost x35 | | Raw felgyőcompost x50 | | Compost after loaded x35 | | Compost after loaded x50 | |
|---|---|---|---|---|---|---|---|---|
| Element | At. ratio | wt% | At. ratio | wt% | At. Ratio | wt% | At. ratio | wt% |
| C | 41.22 | 32.164 | 44.708 | 35.233 | 27.76 | 19.466 | 28.943 | 20.557 |
| N | 12.9 | 11.738 | 10.048 | 9.235 | 10.833 | 8.858 | 10.255 | 8.494 |
| O | 38.163 | 39.667 | 38.043 | 39.936 | 45.348 | 42.358 | 46.147 | 43.66 |
| Na | 0.267 | 0.399 | 0.197 | 0.297 | 0.14 | 0.187 | 0.193 | 0.262 |
| Mg | 0.395 | 0.623 | 0.357 | 0.57 | 0.39 | 0.554 | 0.556 | 0.798 |
| Al | 0.771 | 1.351 | 0.806 | 1.428 | 2.284 | 3.598 | 2.56 | 4.085 |
| Si | 2.759 | 5.035 | 2.552 | 4.703 | 10.304 | 16.895 | 8.456 | 14.044 |
| P | 0.444 | 0.893 | 0.442 | 0.899 | 0.18 | 0.325 | 0.231 | 0.423 |
| S | 0.288 | 0.599 | 0.352 | 0.74 | 0.099 | 0.185 | 0.113 | 0.213 |
| Cl | 0.124 | 0.286 | 0.097 | 0.225 | | | | |
| K | 0.69 | 1.753 | 0.639 | 1.64 | 0.407 | 0.93 | 0.364 | 0.842 |
| Ca | 1.652 | 4.301 | 1.302 | 3.423 | 1.424 | 3.331 | 1.363 | 3.231 |
| Fe | 0.328 | 1.19 | 0.456 | 1.672 | 0.651 | 2.123 | 0.615 | 2.031 |
| Cd | | | | | 0.181 | 1.191 | 0.205 | 1.36 |
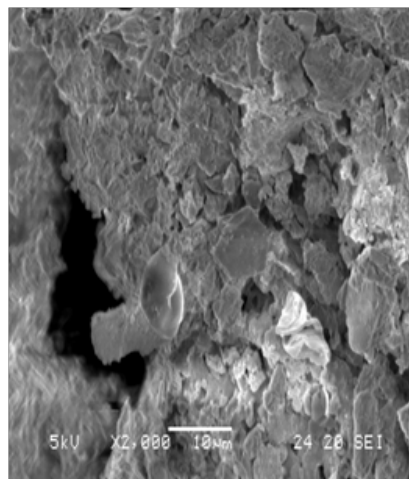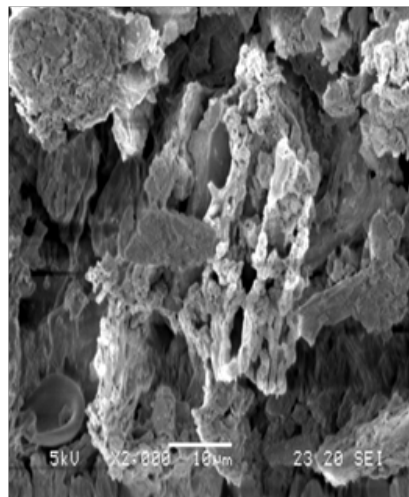
**A**
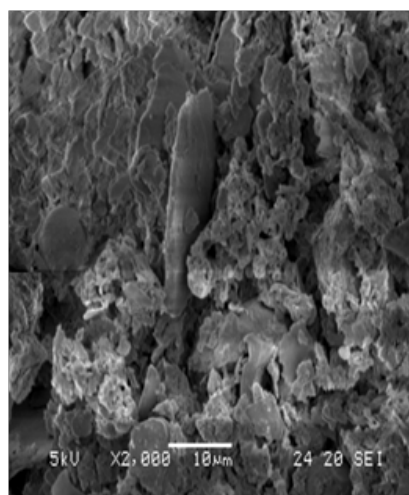


**B**



**C**
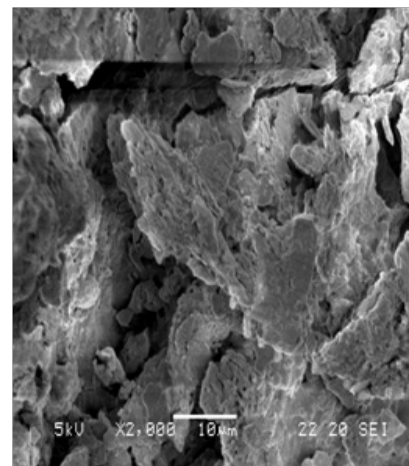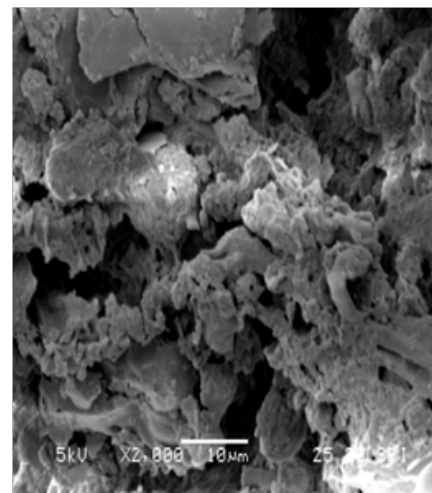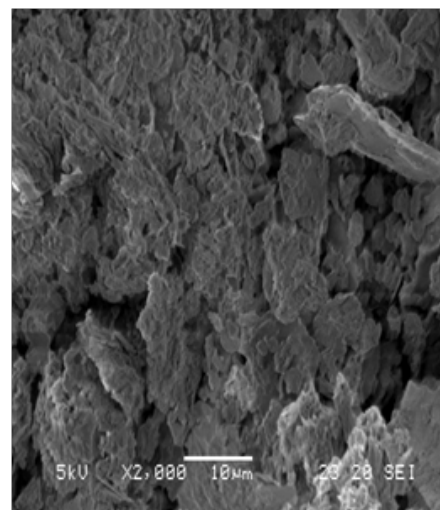
**Figure 1** Scanning electron microscopy (SEM) for the 3 composts loaded with Cd.

**A**

**B**

**C**

**Figure 2** Scanning electron microscopy (SEM) for 3 composts loaded with Cu.

**Fourier transform infrared spectroscopy (FTIR):** While the characteristic peaks of the main organic components are present in all spectra, no significant differences can be observed: the different ratio of the various peaks (indicating different composition from the major organic substances) is probably simply related to the very heterogeneous compost sample as very low amounts of sample (less than 10mg) is required for a pellet. Moreover, there were no major changes such as the appearance or disappearance of peaks was observed in the treated samples when compared to the references. Supposing that the metal binding is mainly due to the ionic interaction of the metal cations with the carboxyl groups in the humic substances, a shift in the ratio of the peaks related to the protonated and dissociated carboxyl groups could indicate the metal binding. However, the peak related to the protonated carboxyl around $1720cm^{-1}$ is not observed even in the reference, probably due to the salt formation occurring with other present cations (such as Na, K etc.), and during the treatment only thechange of the cation occurs; thus the metal binding

has no noticeable effect on the spectra. Figures 5 & 6indicated the peak of 3400 is wide band related to the OH and NH groups and 2930 C-H stretching in the -CH2-groups; 1630 C=O bond (mainly in the carboxyl groups), though the relative wide peak probably indicates the presence of C=C and amide groups; as well, 1430 Related to the O-H and C-O bonds, then 1380 probably CH bonds, although other peaks related to other groups such as nitrate may also be present, which is indicated by the non-steep curve at lower wavelengths. 1070, 1030 Relatively wide band related to the C-O-C bonds invarious polysaccharides, the size and relativity of the two peaks is simply related to the differences in the polysaccharide ratio (such as the starch, cellulose, etc. content; the presence of clay minerals may also effect the band). There are likely more characteristic peaks, which overlap with the most intensive ones mentioned above, thus making their identification difficult in a complex sample like compost, which contains a wide variety of organic compounds.
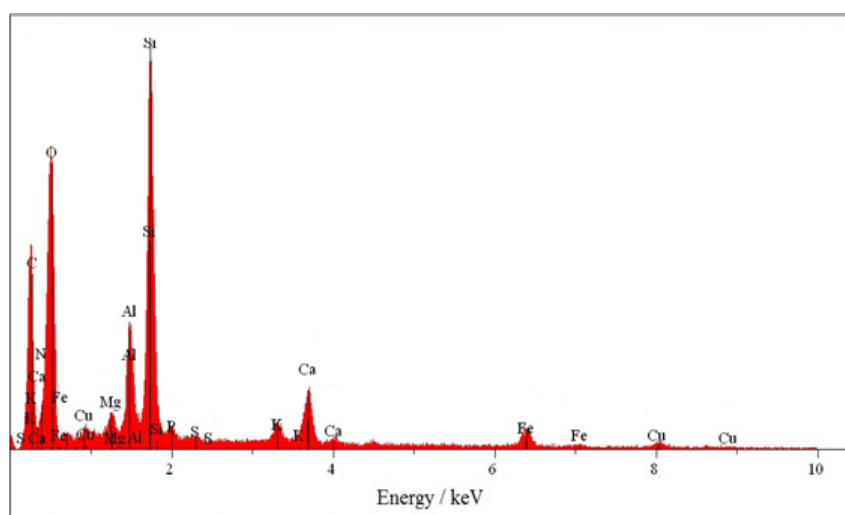


**Figure 3** Energy dispersive X-ray spectroscopy (EDX) for Cu (Felgyőcompost).
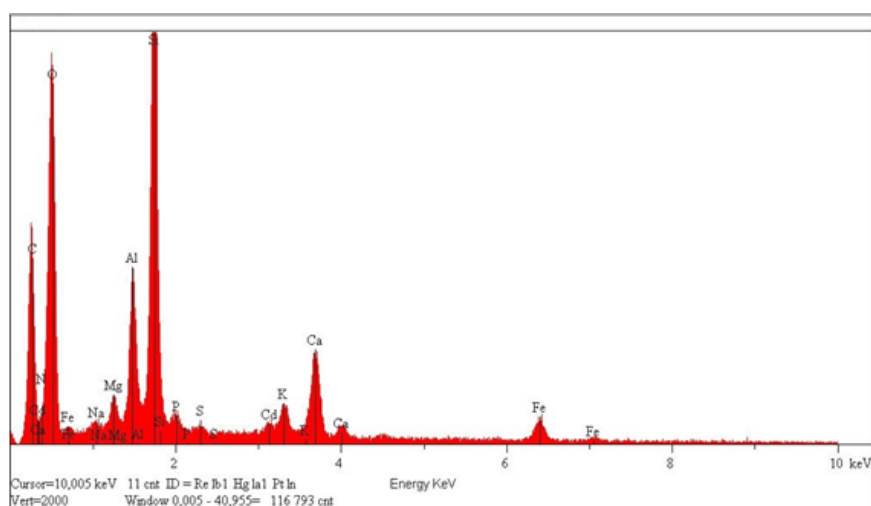


**Figure 4** Energy dispersive X-ray spectroscopy (EDX) for Cd (Felgyő compost).

## Adsorption behaviour in batch experiment

The compost has a significant impact on the uptake of heavy metals in this research. The results obtained are shown in Figures 7 & 8 and in terms of compost effect on the sorption of Cd and Cu from the aqueous solution onto the different sorbents in relation of the metal ions removed percentage. It is clear that Cd and Cu ions were effectively adsorbed in the composts and the maximum adsorption of Cd and Cu ions using compost occurred at 0 to 10000µg/g by 100%. The results in Figures 4 & 7 & 8 show that the equilibrium capacity of cadmium, copper removal by the different adsorbents increased significantly as the of the concentration solution increased. The adsorption capacities of Cu and Cd ions increased rapidly as the concentration value increased, above 10000µg/g the adsorptive capacities of Cd and Cu ions increased, but at a slower rate. Because of the competitive adsorption between hydrogen ion and the heavy metal cation.[9] The adsorption equilibrium were analyzed for Langmuir models using non-linear regression. It was observed that the metal uptaken by composts varied with varying of initial solution concentration and composts as shown in Figures 7 & 8. The cadmium in Figure8 adsorbed was higher in compost Garé then Sioagárd and less in Felgyő, the maximum adsorbed capacity in compost Garé was recorded 40107 while in Sioagrad was 30669 and recorded less amount adsorbed in Felgyő with 26594, the equilibrium constant presented Figure20. 037 at compost Felgyő, 0,027 at compost Sióagárd and less steep at compost Garé. In Figure5. The greater percentage of Cd in the adsorbents was

associated with the residual fractions. The possible explanation for the residual state of adsorbed Cd by compost may be due to the existence of strong chemical bonds between Cd and compost organic matter; organometal complexes exhibited higher stability and reduced the mobility of heavy metal ions.[10] Table 3 is depicted the organic matter content of the 3 composts. Cupper has recorded the highest amount in Figure7 of 44242 adsorbed into compost Garé and 30237 in compost Sióagárd finely in Felgyő compost. Moreover, the content of Cu adsorbed by both composts exceeded Cd, indicating the competitive adsorption advantage of Cu and further illustrated the selective adsorption order. This was consistent with the adsorption order of heavy metals by humus substances.[11] Felgyő was the lowest amount sorbet of cupper with record of 26350. The curves in Figure7 and equilibrium constant K in Table 4 have shown higher steepness in compost Sióagárd 0.05, then Felgyő, 0.04 and lesser in Garé 0.02, respectively. The initial concentration provides an important driving force to overcome all mass transfer resistances of solutes between the aqueous and solid phases. The impact of initial heavy metal concentration on the rate of sorption are shown in Figures 7 & 8, from those figures we can learn that the metal removal differs with varying of initial heavy metal concentration. As shown in Figures 7 & 8 when the initial cations concentration increased, the uptake capacity of red mud increased respectively. A higher initial concentration provided an important driving force to overcome all mass transfer resistances of the pollutant between the aqueous and solid phases thus increased the uptake.[12]
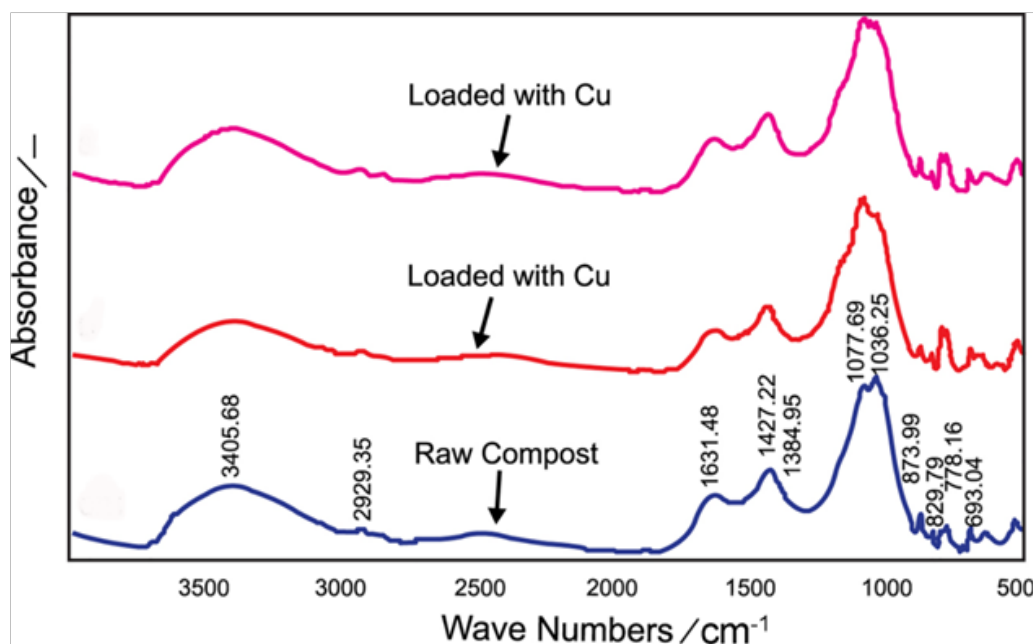


**Figure 5** Fourier transform infrared spectroscopy (FTIR) for Cu (Felgyőcompost).

## Langmuir isotherm

The Langmuir isotherm deals with the unimolecular thick layer of adsorbate upon the surface of a homogeneous adsorbent without having any interactions between adsorbed molecules. Its mathematical form is given by.[13]

$$q_e = q_{max}KC_e/1+KC_e$$

Where $C_e$ is the equilibrium concentration (mg/L), $q_e$ and $q_{max}$ are respectively the amount and maximum amount of metal ion sorbed at equilibrium per unit weight of sorbent (mg/L) and $K_L$ is the equilibrium adsorption constant.
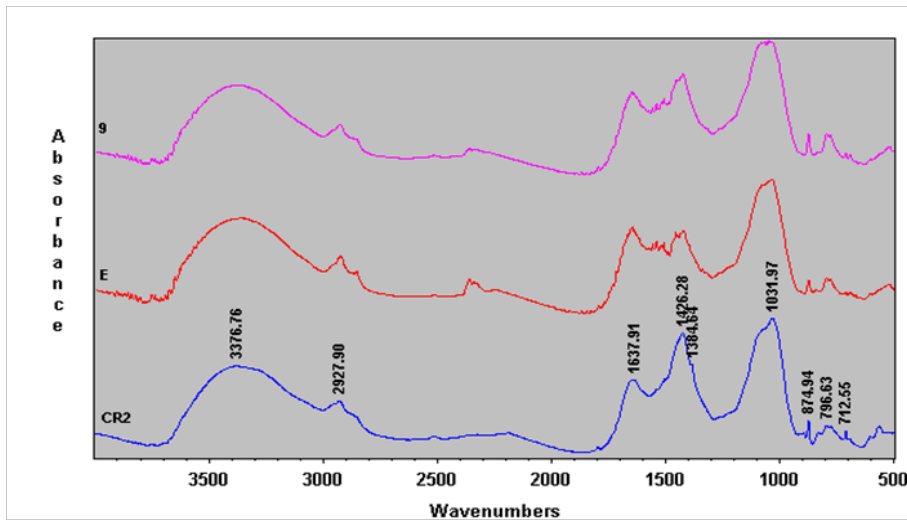
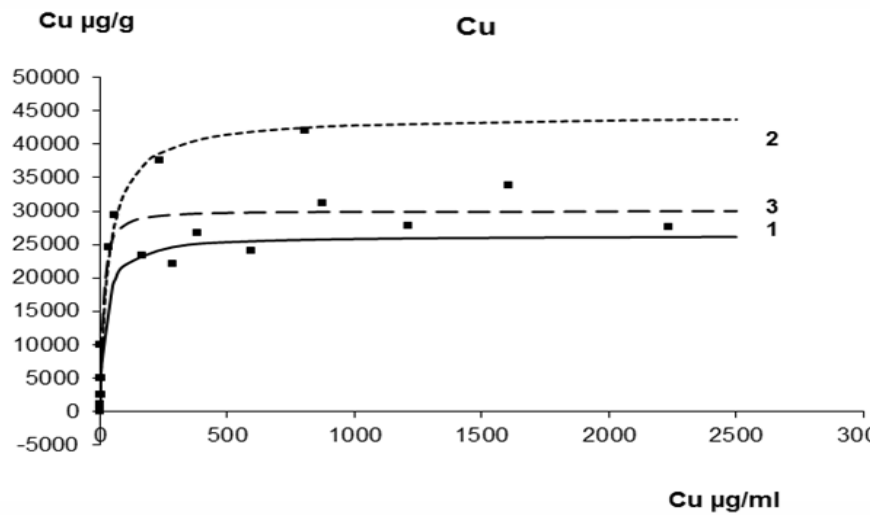**Figure 6** Fourier transform infrared spectroscopy (FTIR) for Cd (Felgyőcompost).



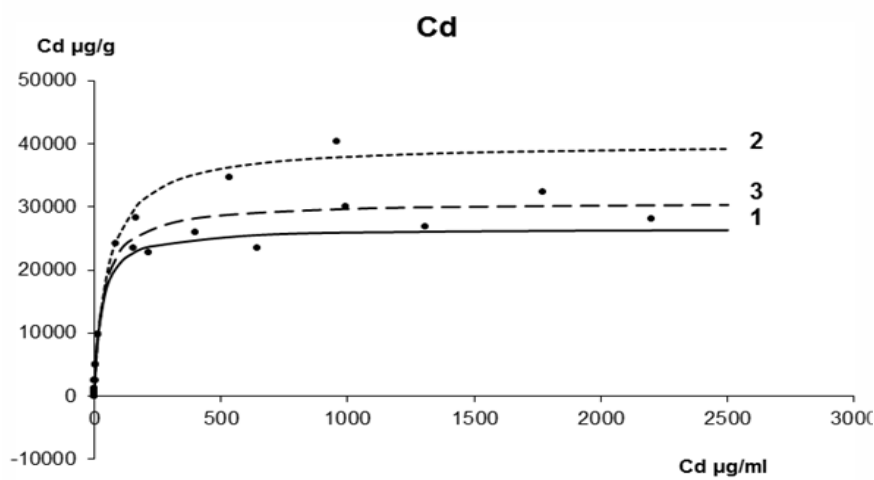**Figure 7** Experimental data of Cu adsorbed by compost 1. 2 and 3 fitted to Langmuir model.



**Figure 8** Experimental data of Cd adsorbed by compost 1. 2 and 3 fitted to Langmuir model.

**Table 3** Content properties of compost materials

| Compost material | Dry matter | $pH_{kCl}$ | $pH_{H2O}$ | OrganicNH$_4$-NNO$_3$-Nsalttotalmatter %mg/kgmg/kg%N %P$_2$O$_5$%K$_2$O% |
|---|---|---|---|---|
| 1.Felgő | 64.3 | 6.0 | 6.6 | 24.717.89421.81.10.70.7 |
| 2. Garé | 46.3 | 5.1 | 5.4 | 50.095.821754.52.63123313453 |
| 3. Sióagrd | 61.7 | 7.0 | 7.3 | 62.216.514611.31.0165549998 |

**Table 4** Langmuir Isotherms for Cd and Cu ($q_{max}$ Maximum Adsorption and $K_L$ Equilibrium Constant).

| Metal | Cd | | Cu | |
|---|---|---|---|---|
| Compost material | $q_{max}g^{-1}$ | $K_L$ | $q_{max}g^{-1}$ | $K_L$ |
| 1. Felgyő | 26594 | 0.037 | 26350 | 0.049 |
| 2. Garé | 40107 | 0.017 | 44242 | 0.028 |
| 3. Sióagárd | 30669 | 0.027 | 30273 | 0.058 |

**Table 5** Removal percentage of Cu and Cd

| $C_0$ | Removal percentage of cadmium | | | Removal percentage of cupper | | |
|---|---|---|---|---|---|---|
| | First compost | Second compost | Third compost | First compost | Second compost | Third compost |
| 50 | 97 | 96 | 97 | 90 | 86 | 86 |
| 100 | 98 | 97 | 97 | 93 | 92 | 92 |
| 250 | 98 | 97 | 98 | 95 | 93 | 96 |
| 500 | 98 | 98 | 98 | 97 | 96 | 97 |
| 1000 | 98 | 98 | 99 | 97 | 97 | 98 |
| 2500 | 99 | 98 | 99 | 98 | 98 | 99 |
| 5000 | 99 | 98 | 99 | 99 | 98 | 99 |
| 10000 | 98 | 98 | 98 | 99 | 99 | 99 |
| 25000 | 91 | 96 | 94 | 88 | 98 | 93 |
| 30000 | 78 | 94 | 86 | 80 | 98 | 87 |
| 40000 | 67 | 86 | 75 | 69 | 94 | 78 |
| 50000 | 56 | 80 | 63 | 55 | 83 | 67 |

## Conclusion

The results of this study suggest that an adsorption system, utilizing compost, can be an economically feasible technique for the treatment of heavy metals in aqueous solution. It was observed that the metal removal varied with varying initial metal concentration. The compost materials used for cleaning the water polluted with Cu²⁺ could adsorb practically 78 to 98%. The FT-IR, SEM-EDX methods were efficient techniques for investigating the physico-chemical characters concerning the compost and the process of adsorption before and after the metal loaded. EDX findings proved that the copper ions were efficiently removed by the adsorbent, the features of Cu²⁺ can be observed on EDX spectrum at different energy values. In addition, compost is very effective and environmentally friendly adsorbent which can be used in wastewater treatment without any chemical treatment. All the 3 compost materials used for cleaning the water polluted with Cd, Cu respectively, could sorb practically the total amount of 100% of heavy metals up to 10000µg/g concentrations. It was observed that the metal removal varied with varying initial metal concentration**.** The cupper shows its vanity by adsorbing into compost Garé, followed by cadmium. The tree heavy metals adsorbed better into Garé compost material rather than Felgyő and Sióagárd. Garé compost has demonstrated outstanding removal capabilities for heavy metals. At common heavy metal pollutions of wastewater the compost materials could be effective sorbent of heavy metal ions. Based on these results, the order of selective adsorption of heavy metals by different composts mainly depends on the chemical nature and the concentration of metal ions. It can be demonstrated that the secondary compost could be used as heavy metal passivation material for the remediation of contaminated soil, and future work should focus on the application of composts as the value-added product for removal of heavy metals.

## Acknowledgements

## Conflicts of interest

Authors declared that there is no conflicts of interest.

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