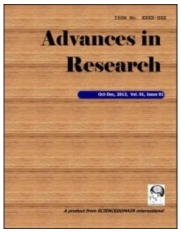


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# Removal of Lead (II) Ions from Aqueous Solution by *Tricholoma terreum:* Kinetics Studies

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***Authors' contributions***

*Author EAO designed the study, wrote the first draft of the manuscript. Authors AOA and ODO managed the biomaterial characterization. Author KKA performed the statistical analysis; while author OA managed the literature search. All authors read and approved the manuscript.*

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## ABSTRACT

Lead and its various compounds have the potency of causing several health problems such as blood disorder, hypertension, renal impairment, neurological damage among others. The removal of Pb (II) ions by *Tricholoma terreum* was performed in a batch biosorption experiments to evaluate the effects of initial concentration of adsorbate, contact time, temperature and solution pH. Maximum biosorption efficiency was obtained to be 11.9 and 14.0 mg/g by untreated and acid-treated mushroom at initial adsorbate concentration of 70 mg/L, contact time of 5 hours, temperature of 60$^{o}$C and solution pH of 2.0. The biosorption data was well fitted by the Ho's pseudo-second-order kinetic model.

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FT-IR characterizations of the mushroom before and after adsorption suggest that carbonyl group, amide and aromatic double bonds probably participated in the adsorption process.

*Keywords: Biosorption; kinetics; Lead (II) ions; pollution; Tricholoma terreum.*

## 1. INTRODUCTION

The whole thing on our planet is interrelated, and while the nature supplies us with valuable environmental services exclusive of which we cannot survive, we all depend on each other's dealings. These dealings between man and its environment on daily basis has led to the obliteration and contagion of the environment. The contagion and obliteration of the environment is called pollution. Environmental pollution occurs when the environment can no longer process harmful by-products of human activities such as oil spillage, mining, domestic waste, industrial effluents etc. The discharge of untreated and partially treated metal containing effluents into the waterway and the continuous mining activities are the major sources of contamination in the aqueous system owing to the presence of heavy metals [1]. One of such heavy metal is lead. Lead is a poisonous element causing several kinds of diseases. Among the health problems associated with lead include kidney damage, reproductive system and cardiovascular impairments, enzymes inhibition, anemia and death [2]. Lead is commonly emitted from materials such as incomplete burning of fuels, photographic materials, batteries, printing materials, pigments and paint materials [3,4]. According to Rani et al. [4], petrol combustion globally contributes an estimated amount of 60 % of total lead emission.

In the recent years, the research for the removal of toxic metals from aqueous solution has focused on new technologies rather than conventional methods such as chemical precipitation, ion-exchange, solvent extraction and membrane processes which are generally considered not only to be expensive but are also inefficient in the removal of these toxicants from contaminated environment [5,6].The biosorption to materials of biological origin has been proposed as an alternative to conventional methods for the economic removal of toxicants [7]. Biosorption is the processes in which microorganisms or agricultural wastes are used to remove and recover heavy metals and other toxic materials from aqueous solution. Among the fungi that has been widely used for heavy metal removal include *Bacillus subtilis* [8], *Trichoderma longibrachiatum* [9], *Laminaria japonica* [10], *Sargassum sp.* [3], *Aspergillus niger RH 17* and *A. niger RH 18* [4], *Bisidiomycetes* [11], *Trichodema* and *Agaricus* [12]. The removal of heavy metals by microbial biomasses from unhygienic surroundings depends on factors such as the concentration and availability of metal, microbial species, age of mycelia, temperature, pH, nature of medium, types of metal and availability of nutrient. According to Lucinal et al. [13], biomaterials like fungi have been proven to be more efficient and economical for the removal of toxic metals from dilute aqueous solution because of their filamentous morphology and high percentage of cell walls. Padma and Bajpai [12] reported that *Trichodema* reduces chromium concentrations in solution by 97.39% and *Agaricus* removed 100% of the metal at pH of 6.6. The cell walls of microbial biomasses is believed to be composed of proteins, polysaccharides, lipids which have abundant metal binding groups such as the carbonyl, sulphate, hydroxyl, amine, phosphate which are responsible for the metal removal.

Mushroom is the freshly, spore –forming fruiting body of a fungus which is typically produced above ground on soil or on its food source. Mushroom is a low calorie food which is usually cooked before eaten or can also be eaten as raw and as garnish to a meal. Edible mushroom are good source of vitamin B and the essential elemental minerals such as selenium, potassium and copper. The carbohydrates, calorie and fat contents are low with the absence of vitamin C. *Tricholoma terreum* is commonly known as the grey knight or dirty *Tricholoma*. It is a grey-capped edible mushroom of the large genus *Tricholoma*. It is a fruiting mushroom consisting of a cap (pileus) with a spore-bearing part and stipe (stem, stalk) and with a soft taste but need to be eaten within a day or two of picking since the gills and flesh yellow spoil quickly. The aim of this work is to examine the ability of *Tricholoma terreum* for the removal of lead (II) ions from aqueous solution. Effects of factors such as solution pH, initial concentration of adsorbate, temperature and contact time were investigated. The aim of the acid-treatment is to extract soluble organic compounds, improve cellulose hydrolysis, remove impurities and increase surface porosity or surface area. The adsorption data were modeled into different kinetic models.

## 2. MATERIALS AND METHODS

### 2.1 Materials

All the glassware used were thoroughly washed with detergent and tap water then rinsed with distilled water and thereafter soaked in 10 % nitric acid (Oxford Laboratory, Indian) for 1 hour after which they were rinsed with distilled-deionised water and dried.1000 mg/L stock solution of $Pb(NO_3)_2$ (Merck, Germany) was prepared. Working solutions of initial concentrations of 15,20,30,40,50,60,70,80,90 and 100 mg/L were then prepared from the stock solution using the dilution principle.

### 2.2 Biomass Preparation

The *T. terreum* was sampled locally in a palm oil mill village at Kajola in Odigbo Local Government Area of Ondo State, Nigeria and it was washed with distilled water to remove dirt. The water contents was removed by keeping the mushroom on a filter paper and thereafter, it was cut into smaller pieces and sun dried for 24 days followed by oven drying which was done at a temperature of 70ºC for 3 hours. Subsequently, the biomass was ground on an Agate mortal pestle and sieved through a mesh size of 250 µm size. The acid-treated mushroom was prepared by transferring about 50g of the raw mushroom into 250 ml of 0.1M HCl and allowed to stay for 24 hours. The fungi biomass was then removed washed with distilled-deionised water and dried in an oven at 80ºC for 4 hours. The raw biomass was referred to as untreated mushroom (UTM) while that which was treated with acid was referred to as acid-treated mushroom (ATM). The samples were kept in sample bottles prior to analysis.

### 2.3 Batch Kinetics Experiments

The batch experiments were performed in 250 ml conical flask with a working volume of 15 ml of lead (II) ions concentration. 0.5 g of the biosorbent was added to the 15 ml solution of the lead (II) ion and agitated at a speed of 150 rpm on a temperature controlled shaker for 5 hours at ambient temperature. Samples were then collected, filtered and the filtrate was analysed for lead (II) ions contents using Buck Scientific model 210VGP Atomic Absorption Spectrophotometer. This procedure was repeated by using different concentrations of lead (II) ions, pH, contact time and temperature. Each sample was analysed in triplicate and a mean concentration was calculated by averaging. Controls were also run by adopting same method but without the addition of the biosorbent. The amount of lead (II) ions adsorbed ($Q_e$) in mg/g were calculated and the values were multiplied by 100 to put them on percentage basis.

$$Q_e = \frac{C_o - C_e}{m} V \quad \text{(Eq.1)}$$

$$Biosorption\ Efficiency\ (\%) = \frac{C_o - C_t}{C_o}\ X\ 100 \quad \text{(Eq. 2)}$$

Where $C_o$ (mg/L), $C_e$ (mg/L) and $C_t$ (mg/L) are the initial metal concentration, equilibrium concentration and concentration at time *t*, *V* is the solution volume in *L* and *m* is the mass of the dry biomass used in g.

## 2.4 FT- IR Characterization of Biomass

Vibrational frequencies of the dried adsorbent (mushroom) were obtained before adsorption and after adsorption of lead from FT-IR transmission by KBr method. The background measurement was performed with homogenized pellet of grinded KBr powder pressed with SHIMADZU MHP-1 mini hand press. About 5% of the samples in KBr were also prepared into homogenized pellets. Suitable pellets were used for the FT-IR measurement in the percentage transmittance mode. The scans were done in the mid IR region of 400-4000 $cm^{-1}$ with 45 scan repeat using SHIMADZU 8400S FT-IR instrument.

# 3. RESULTS AND DISCUSSION

## 3.1 FT-IR Characterization of Biomass

Fig. 1. Shows the FT-IR spectra of the dried adsorbent before adsorption (mushroom only) and after adsorption (mushroom+Pb) in the range 4000–400 $cm^{-1}$. A wide peak in the spectra between 3600 and 3000 $cm^{-1}$ is characteristic of N – H stretching of N – substituted amide. The medium absorption of 2920 and 2362 $cm^{-1}$ are vibrational stretching of C-H and N-H groups respectively. The Peak at 2850 is the C–H stretching of aldehyde, while the carbonyl (C=O) stretching of ketone, esters, lactones, quinines and/or carboxylic acids is reported at 1739 $cm^{-1}$. Also the aromatic C-H bending peaks are found between 800 and 400 $cm^{-1}$ Olukanni et al. [14]. The two spectra are similar with slight shift in some characteristic absorbance peaks. The approximate absorption bands in the adsorbent shifted at 1735, 1629,1508,1078 and 1232 $cm^{-1}$ when it adsorbed Pb as against the absorption at 1739, 1624,1518,1244 and 1033 $cm^{-1}$ without Pb on it. This suggests that carbonyl group, amide and aromatic double bonds probably participated in the adsorption.The disappearance of most of the peaks between 1520 and 1150 $cm^{-1}$ in the adsorbent+Pb spectrum shows that the aromatic double bonds are involved in the adsorption. This is confirmed by the increase in the intensity of the C-H bending peaks of the same spectrum; particularly those of the 777 and 457 $cm^{-1}$ peaks.

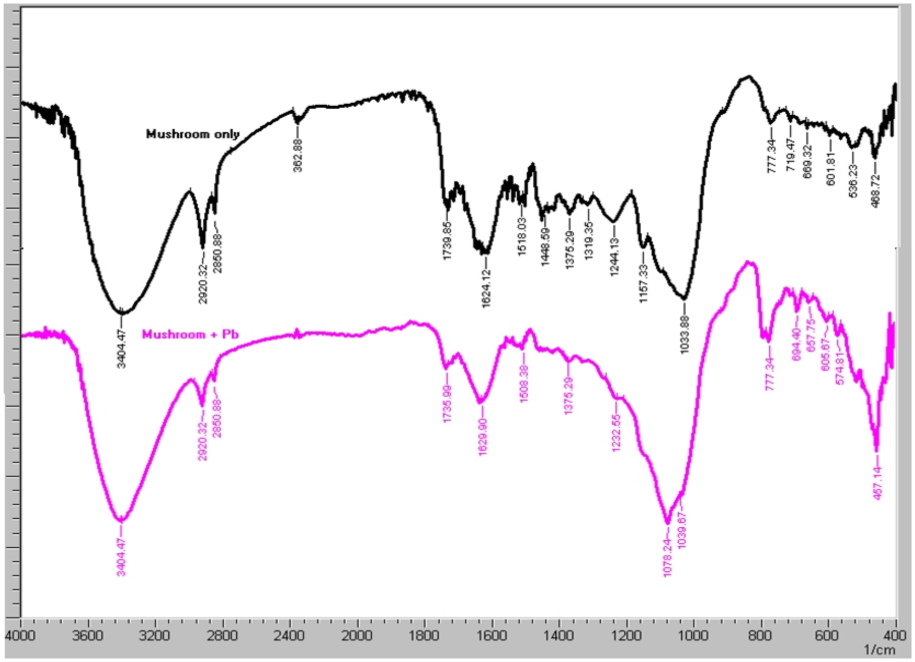


**Fig. 1. FT-IR Spectra of mushroom before adsoprion and after the adsorption of lead (II) ions**

## 3.2 Effects of Initial Adsorbate Concentration

The initial metal concentration is a significant factor in determining the adsorption potency of a biomass. The effect of initial concentration of the adsorbate was evaluated at different concentrations of 15,20,30,40,50,60,70,80,90 and 100 mg/L at a temperature of 60ºC, contact time of 5 hours and pH of 2.0 which is shown in Fig. 2. As revealed in the figure, the amount of Pb (II) ions adsorbed increases with increase in the initial concentration of the adsorbate. An initial adsorbate concentration of 70 mg/L was however enough to achieve a maximum adsorption of 11.4 and 14.0 mg/g by untreated and acid-treated mushroom respectively. Beyond the concentration of 70 mg/L, there was negligible increase in the amount of Pb (II) ions adsorbed. Thus, an initial metal concentration of 70 mg/L was selected for subsequent experiments. Negligible increase in the adsorption of Pb (II) ions which was observed at higher initial metal concentrations above 70 mg/L may be due to the saturation on the surface of the biomass. The comparison of the results of this work with Nale et al. [15], Adeogun et al. [9] and that of Emmanuel and Veerabhadra, [16] shows a better agreement.

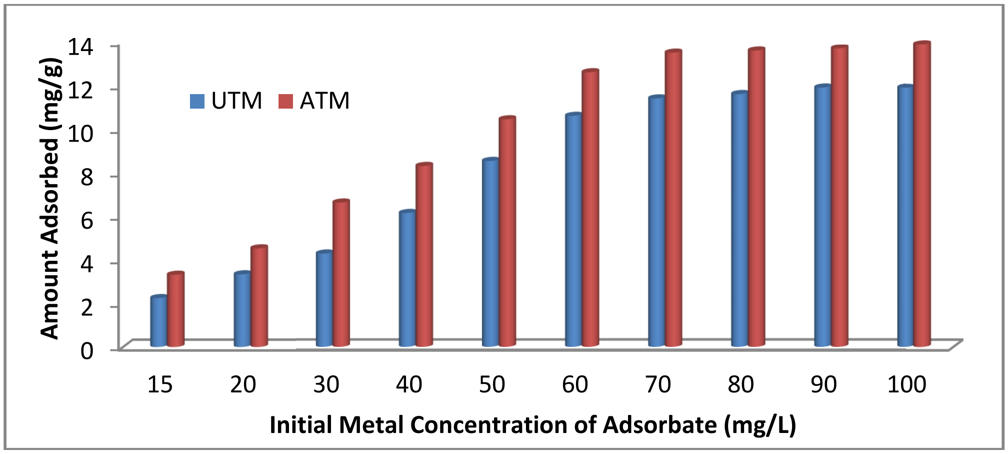


**Fig. 2. Effects of initial concentrations of adsorbate in mg/L at a pH of 2.0, contact time of 5 hours, biosorbent concentration of 0.5 g and temperature of 60ºC**

### 3.3 Effects of Contact Time

The effect of contact time on the efficiency of biosorption of Pb (II) ions by untreated and acid-treated mushroom is shown in Fig. 3. As seen, the efficiency of uptake of Pb (II) ions by both biomasses increases with the contact time. The results showed that a contact time of 5 hours is enough to attain equilibrium. Above this time, there was no further appreciable adsorption of Pb (II) ions by the biomasses. The adsorption process was however very rapid at the initial stage and slow down as the reaction approached the equilibrium stage. The rapid adsorption process which was observed at the initial stage may be due to the rapid accumulation of Pb (II) ions on the pore sites of the *T. terreum* as a result of the presence of vacant sites on its surface. These vacant sites become occupied by Pb (II) ions as the reaction proceeds thereby making the penetration of more Pb (II) ions into the interior of the biosorbent difficult. Similar observation was reported by Sangi et al. [17] and Qaiser et al. [18].

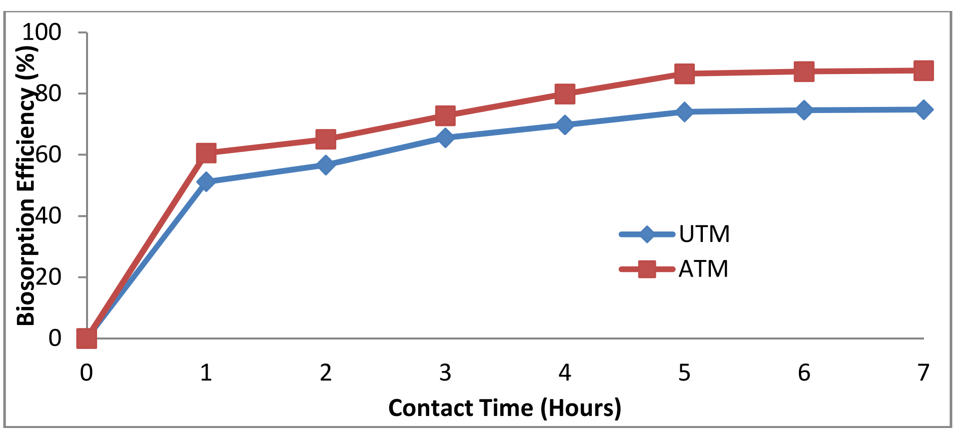


**Fig. 3. Biosorption efficiency (%) of *T. terreum* versus contact time (hours) at initial metal concentrations of 70 mg/L, pH of 2.0, biosorbent concentration of 0.5 g and temperature of 60ºC**

## 3.4 Effects of Temperature

Different temperature ranges between 25 to 70ºC were selected to investigate the effects of temperature on the biosorption efficiency of *T. terreum* which is shown in Fig. 4. As depicted in Fig. 3, the biosorption efficiency of *T. terreum* increases with an increase in the temperature of the system to a certain temperature of 60ºC before showing a decrease. The biosorption efficiency increased from 54.3 to 89.9% when the temperature was increased from 25 to 60ºC before showing a decrease to 79.5 at a temperature of 70ºC when untreated mushroom was used. Upon treatment of the biomass with acid, the biosorption efficiency increased from 56.6 to 94.6% on increasing the temperature from 25 to 60ºC before it show a decrease to 88.6% at a temperature of 70ºC. Qaiser et al. [18] observed that higher temperature caused a change in the biomass texture which reduces the biosorption capacity. This may be the reason why a decrease in the biosorption efficiency was observed at a higher temperature above 60ºC in this work.

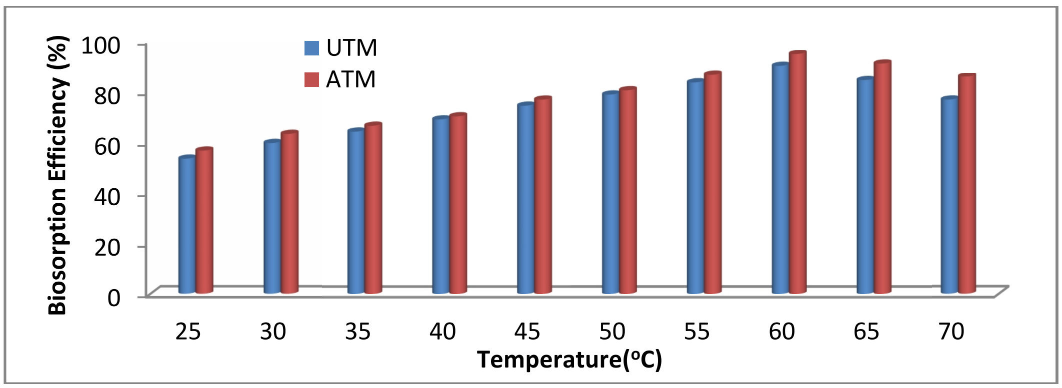


**Fig. 4. Biosorption efficiency (%) of *T. terreum* versus temperature (ºC) at initial metal concentrations of 70 mg/L, pH of 2.0, biosorbent concentration of 0.5 g and contact time of 5 hours**

## 3.5 Effects of Solution pH

Fig. 5. shows the plots of the effects of pH on the biosorption efficiency of *T*.terreum. The results showed that the biosorption of Pb(II) ions by both UTM and ATM was greatly affected by the solution pH which resulted in a decrease in the biosorption efficiency when the solution pH increases into the basic medium. The absorption efficiency decreased from 70.3 to 45.4% for UTM and from 86.5 to 51.5 for ATM on increasing the solution pH from 2.0 to 7.0. Maximum biosorption was obtained at an acidic pH of 4.0 for both biomasses. According to Adeogun et al. [9] and Wang et al. [18], the surface walls of most biomasses contained functional groups such as amines, hydroxyl, carbonyl, carboxylic acids, sulphates which are positively charged at low pH which hindered the adsorption of the metal ions by the biomass, but with increase in the pH, the surface become negatively charged which enhance the adsorption of the metal ions by biomass. The increase in the biosorption efficiency at higher pH may be due to the charged on the cell walls of the biomass at higher pH values. The results from this work is in agreement with what was obtained by Adeogun et al*.* [9], Wang et al. [19] and Zhao et al. [20].

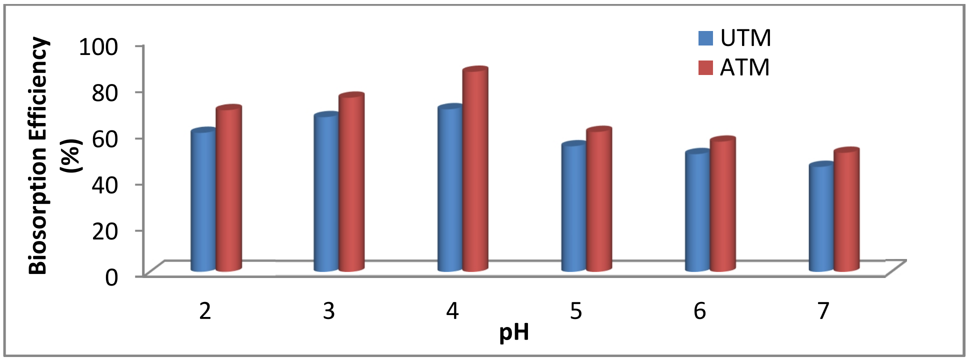


**Fig. 5. Biosorption efficiency (%) of *T. terreum* versus pH at initial metal concentration of 70 mg/L, contact time 5 hours, biosorbent concentration of 0.5 g and temperature of 60ºC**

### 3.6. Analysis of Kinetics Data

Kinetic analysis can be performed by model-fitting. Many models have been proposed for kinetics studies, and models are developed based on certain mechanistic assumptions. Largergren's first-order and Ho's pseudo-second-order models were applied to describe the adsorption data. The Largergren's –first- order model equation is given as:

$$In(Q_e - Q_t) = InQ_e - \mathrm{k}_1 t \qquad (Eq.3)$$

Where $Q_e$ and $Q_t$ (mg/g) are the amount of Pb (II) ions adsorbed by *T. terreum* at equilibrium and at time in hours respectively, $\mathrm{k}_1$ ($h^{-1}$) is the rate constant of first order. The linear plots of $Q_e - Q_t$versus *t* which is shown in Fig. 6. were used to estimate the values of $\mathrm{k}_1$ and $q_e$ which were obtained from the slopes and intercepts of the plots. The physical parameters are presented in Table 1. The values of the correlation coefficients ($R^2$) were lower when compared with that of Ho's pseudo-second-order model. Also, values of the experimental adsorption capacity ($Q_e$.exp.) are not in agreement with the calculated adsorption capacity ($Q_e$ cal.). This suggest that despite the closeness of the values of the correlation coefficients to unity, the adsorption of Pb (II) ions by both UTM and ATM do not fit well into the Largergren's-first-order kinetic model. The expression for the Ho's pseudo-second-order kinetic model is given as:

$$\frac{t}{Q_e} = {}^{1}/_{k_2}\, Q_e^2 + {}^{1}/_{Qq_t}\, t \qquad \text{(Eq.4)}$$

Where $k_2$ ($gmg^{-1}h^{-1}$) is the rate constant of second-order, $q_e \; and \; q_t$ (mg/g) are the amounts of Pb (II) ions adsorbed at equilibrium and at time *t* in hours. The linear plots of $\frac{t}{Q_e}$versus *t* which is shown in Fig. 7. were used to estimate the values of $Q_e$ and $k_2$ which were obtained from the slopes and intercepts of the plots and their physical parameters are shown in Table 1. For the second-order model, the values of the correlation coefficients ($R^2$) which were obtained from the plots ranged between 0.964 and 0.998 which are closed to unity. Also, on comparing the values of the experimental adsorption capacity ($Q_e$ exp.) with the calculated adsorption capacity ($Q_e$ cal.), a closed relationship was observed. This further suggests that the adsorption of Pb (II) ions by both biomasses is governed by the Ho's pseudo-second-order model.

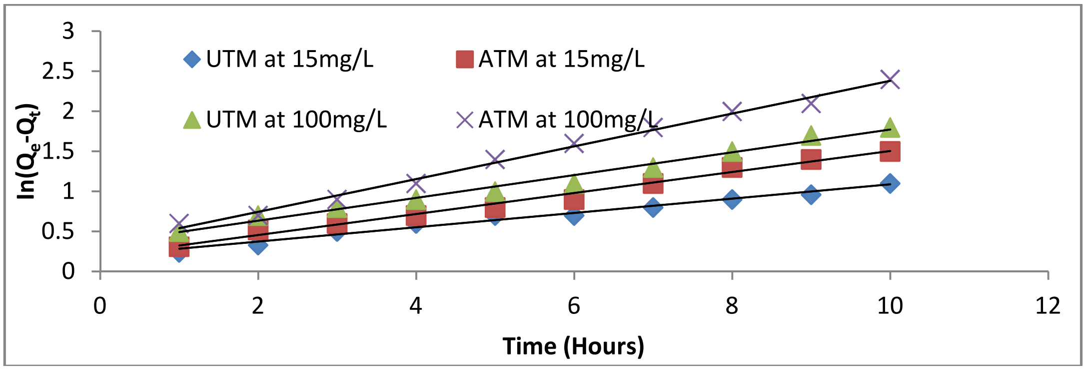


**Fig. 6. Largergren's first-order kinetic model for the biosorption of Pb (II) ions by untreated and acid-treated *T. terreum* at initial concentrations of 15 mg/L and 100 mg/L**

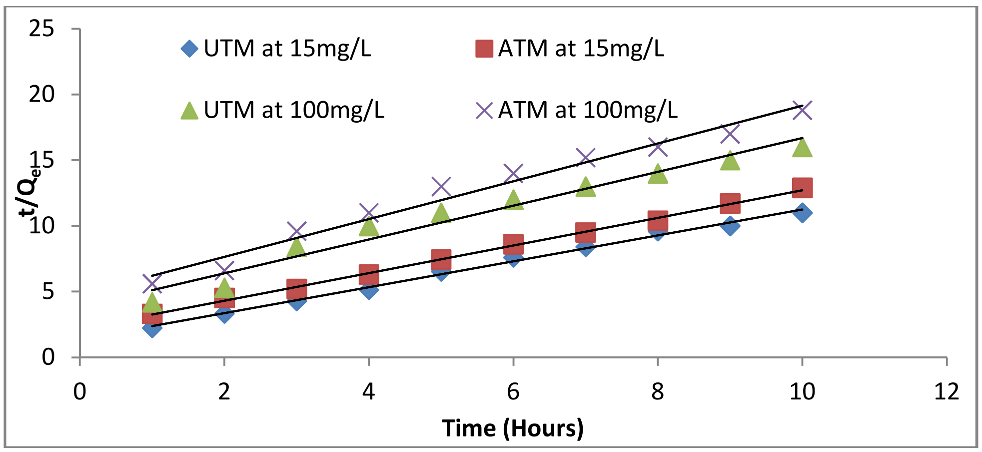


**Fig. 7. Ho's pseudo-second-order kinetic model for the biosorption of Pb (II) ions by Untreated and acid-treated *T. terreum* at initial concentrations of 15 mg/L and 100mg/L**

**Table 1. Kinetic parameters of first-order and second-order kinetic models**

| | | First-order Kinetic | | | | Second-order kinetic | | |
|---|---|---|---|---|---|---|---|---|
| **Biomass** | **$C_o$ (mg/L)** | **$Q_e$(exp) (mg/g)** | **$k_1$($h^{-1}$)** | **$Q_e$(cal.) (mg/g)** | **$R^2$** | **$k_2$ (gmg$^{-1}$h$^{-1}$)** | **$Q_e$(cal) (mg/g)** | **$R^2$** |
| **UTM** | 15 | 2.24 | 0.089 | 0.191 | 0.979 | 0.73 | 3.42 | 0.994 |
| **ATM** | 15 | 3.13 | 0.130 | 0.190 | 0.989 | 0.26 | 2.51 | 0.998 |
| **UTM** | 100 | 11.9 | 0.142 | 5.346 | 0.983 | 0.45 | 10.11 | 0.964 |
| **ATM** | 100 | 15.8 | 0.204 | 7.330 | 0.993 | 0.21 | 17.13 | 0.975 |

Table 2 shows the comparison of the adsorption capacity of *T. terreum* with other biosorbents from literature. As shown in the Table, *Tricholoma T*. has good potential for the removal of lead (II) ions from aqueous solutions.

**Table 2. Comparison of the adsorption capacity of *T. terreum* for the adsorption of Pb (II) ions with other biosorbents**

| Biomass | $Q_e$(mg/g) | References |
|---|---|---|
| *T. Longibrachiatum* | 71.0 | [9] |
| *Ficuss religiosa* leaves | 37.46 | [21] |
| *Aspergilus niger* | 10.1 | [22] |
| Caulerpa lentillifera | 29.0 | [23] |
| Untreated *T. terreum* | 11.9 | This present work (as published) |
| Acid-treated *T. terreum* | 15.8 | This present work (as published) |
| *Parmelina tiliaaceae* | 75.8 | [24] |
| *Aspergillum niger* | 32.6 | [25] |

## 4. CONCLUSION

The efficiency of *T. terreum* to remove Pb (II) ions from aqueous solution was investigated. Both untreated and acid-treated forms of the biomass were deployed to assess the effects of factors such as contact time, initial adsorbate concentration, temperature and solution pH. The results revealed that the biosorption efficiency of *T. terreum* depend greatly on the various factors examined. Maximum adsorption capacity of untreated and acid treated mushroom was obtained to be 11.9 and 15.8 mg/g respectively. Also, the acid-treatment of the biomass before adsorption enhanced the biosorption efficiency of the biomass. FT-IR characterizations of the mushroom before and after adsorption suggest that carbonyl group, amide and aromatic double bonds probably participated in the adsorption process. Thus, it can be concluded that *T. terreum* is a good material for the removal of Pb (II) ions from aqueous solution.

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## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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